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# CHEMICAL NEWS

AND

## JOURNAL OF PHYSICAL SCIENCE

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### CONTENTS.

| ARTICLES:—                                                                                                                                     | PAGE |
|------------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Action of Radium, Röntgen Rays, and Ultra-violet Light on Minerals, by George F. Kunz, Ph.D., A.M., and Charles Baskerville, Ph.D., F.C.S. | 1    |
| Potable Waters in South-West Lancashire, by Prof. J. Campbell Brown, D.Sc.                                                                     | 6    |
| The Solubility of Hydrated Sulphate of Lime in Solutions of Sea-salt, by Alex. d'Anselme                                                       | 9    |
| Colorimetric Estimation of Bismuth, by Paul Planès                                                                                             | 10   |
| New Designs for Chemical Balances, by J. S. Lumsden, D.Sc., &c.                                                                                | 11   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                          | 12   |
| MISCELLANEOUS                                                                                                                                  | 12   |

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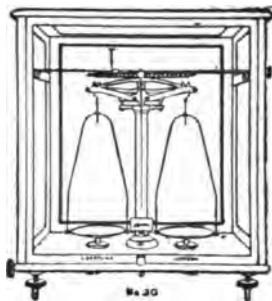
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# THE CHEMICAL NEWS.

VOLUME LXXXIX.

EDITED BY SIR WILLIAM CROOKES, F.R.S., &c.

No. 2301.—JANUARY 1, 1904.

## THE ACTION OF RADIIUM, RÖNTGEN RAYS, AND ULTRA-VIOLET LIGHT ON MINERALS.\*

By GEORGE F. KUNZ, Ph.D. A.M.,  
Honorary Curator of Gems and Precious Stones, American Museum  
of Natural History, and Special Agent for the United States  
Geological Survey,

and  
CHARLES BASKERVILLE, Ph.D., F.C.S.,  
Smith Professor of Chemistry and Director of the Laboratory,  
University of North Carolina.

THE purpose of this paper is to recount the results of our investigations as to the conduct of the gems and gem-material of the Tiffany-Morgan collection under the influence of Röntgen rays, ultra-violet light, and emanations of radium preparations. At the same time, through the courtesy of the American Museum of Natural History, we were permitted to make a careful study of the action of ultra-violet light upon the materials in the handsome Morgan-Tiffany and Morgan-Bement collections. These collections undoubtedly are the most complete authenticated minerals and gems on exhibition in the United States. The fluorescence and phosphorescence resulting from the action of ultra-violet light upon about 13,000 verified minerals were carefully observed. In addition to the above, we had an opportunity to submit selected stones from about 15,000 British Guiana diamonds, and two particularly handsome diamonds (one being a tiffanite), and several carbonadoes, to these influences, the products of the most modern scientific investigations. Our first mention of this investigation was given briefly in *CHEMICAL NEWS*, vol. lxxviii., p. 263.

As there is no uniform meaning accepted for the terms "fluorescence" and "phosphorescence," in the outset we wish to emphasise our interpretation. Jackson would have them meaning the same. Perhaps they are due to the same phenomena; but in this paper by fluorescence we mean a luminosity, more usually evidenced by a play of colour, lasting only during the direct influence of the exciting agent. By phosphorescence we mean the emission or propagation of ethereal stresses, which affect the optical centres, producing light, white or coloured, which persists after the removal of the cause. Substance may therefore be both fluorescent and phosphorescent.

The radium preparations of the highest activity used in these investigations became the property of the American Museum of Natural History through the liberality of Mr. Edward D. Adams, a member of the Board of Administrators of the Museum and of the New York Academy of Sciences. His gift of the necessary funds was applied to the purchase

\* Extracts from Paper presented before the New York Academy of Sciences, October 6, 1903.

of one portion of radium bromide of 300,000 activity, and another of 1,800,000; uranium being taken as the standard at 1. The preparations were obtained from the Société Centrale des Produits Chimique at Paris. Unfortunately, although the order was authorised and material assured, it has been impossible to obtain the bromide of the highest strength in time for presentation at this meeting. The results here announced have to do with the radium of 300,000 and of 7000 activity (chloride), and 240 (chloride), and of 100 radium barium carbonate. The compounds of lower activity were purchased by the authors.

Thorium dioxide does not become luminous in contact with the radium preparations we have been able to obtain, although Elster and Geitel state that a thorium oxide screen shows scintillating fluorescence even after positive electrification, similar to the zinc blende.

Why certain rays either alone or through their influence upon the surrounding media present the order of magnitude of visible light waves, we shall not undertake to say. It is quite evident, however, that particular substances, like the diamond, willemite, kunzite, &c., possess the power of "stepping up or down," as it were, the ethereal stresses propagated by the radium, so that visible rays result, or the bombardment of electronic emanations produce such an effect. The luminosity cannot be attributed solely to the  $\alpha$ -rays, or helium, as thorium oxide does not respond (at least visibly to the eye unaided by magnifying lenses), unless it so happens that the helium exists in one radioactive body in a different form from that in the other. Further, we have tested a number of helium-bearing minerals, and none responded to the strong radium bromide. It appears that the luminosity of such substances so variable in their construction as those mentioned above may be accounted for in a measure by the physical explanations adverted to; yet the striking fact that four zinc compounds of totally different composition, willemite (zinc ortho-silicate), zinc sulphide, zinc oxide, and kunzite (which contains a fraction of a percent of zinc), all respond in the most pronounced manner to radium emanations, could indicate the presence of a new element, a "radium-foil," as it were, or some unusual combination of known chemical integers, which synchronise with the activities of this unique body. One is immediately reminded of the actinium Phipson announced in the eighties as being present in zinc white. (This is not to be confounded with Debiere's actinium, resembling titanium, announced in 1898).

Selected gems were mounted in paraffin blocks held in wooden frames, obtained by pouring the hydrocarbon, after melting in a water-bath, into especially constructed boxes about 18 x 27 c.m. and 1.5 c.m. deep. The wooden bottoms attached to the frames by screws were readily removed. Each "plate" was numbered by placing small

wire nails on the ceresine. Where gems of the same class varied in colour they were arranged according to the spectrum as far as practicable. Comparisons as to colour effects could thus be had, but of course no comparison of penetrative effects, as the stones were of variable thickness and were all photographed in place.

These plates admitted of a careful comparative examination of the gems when subjected to the bombardment of the Röntgen rays produced by a Queen's self-regulating Crookes tube with a twelve-inch spark coil. Many radiographs were also obtained with variable exposures, the iron markers adverted to, through their impenetrability, serving to identify the plates.

Perhaps no such complete collection has been studied in this manner, although Doelter in 1896 published a most interesting study of the conduct of some sixty-five minerals and other precious stones when subjected to the Röntgen rays. During the same year, J. B. Cochrane published a very practical paper on the testing of precious stones by the X-rays. Without giving details, it may be said that their observations were verified, and, in general, it was learned that the penetration of gem material by the X-rays is a matter of degree rather than kind, and sharper contrasts were obtained with certain precious stones when they were surrounded by metals, like gold or lead, than was had when they were radiographed alone. The observations of the late Professor Ogden Rood on the deflection of X-rays by crystal or cut faces were verified.

J. E. Burbank, in 1898, published a work on Mineral Phosphorescence produced by X-rays. Of all the substances tried, he found "in general minerals containing ores of the metals are non-phosphorescent," and that fluorite and calcite seemed most suitable for experimentation. J. Trowbridge states:—"By them (X-rays) an electrical charge is communicated to fluorescent and phosphorescent substances. The resulting electrical energy, in being dissipated (by heat), produces the phenomenon of light" (see below). Thomas A. Edison directed the Röntgen rays upon some eighteen hundred chemical compounds, artificial and natural, seeking fluorescent screens. Later (1900), Bary examined the conduct of a number of salts under the influence of Röntgen rays. "Those which become fluorescent belong, with the exception of uranium, to the families of the alkalis and alkaline earths. Similar results were obtained by exposure to the radiations from a radio-active metal supplied by Curie."

Robert Boyle, in 1663, appears to have been the first to have made a truly scientific examination of the phosphorescence of diamonds, although the alchemist, Albertus Magnus, in the thirteenth century, said he had seen a diamond which glowed when it was put in water. Bernouilli remarked that when a diamond is rubbed on gold it becomes luminous "like a burning coal excited by the bellows," as Draper put it. "A light, too, that cannot be extinguished by water, and yet so ethereal and pure that it can set nothing on fire," attracts the scientific imagination as much to-day as it did then.

Two hypotheses were offered in the eighteenth century in explanation of phosphorescence:—

1. Lemery, in 1709, maintained that phosphorescent bodies act like sponges to light, absorbing it and retaining it by so feeble a power that very trivial causes suffice for its extinction.

2. Du Fay, in 1735, upheld that it resulted from actual combustion taking place in the sulphureous parts of the glowing body. He first noted the substance requires previous exposure to light; it glows in the dark with decreasing luminosity. Du Fay also observed the effect of interposing coloured glass between the phosphorescent body and the light.

In 1859, J. H. Gladstone, exhibited diamonds strongly fluorescent in the sunshine. Silvanus Thompson used one of these diamonds in 1896 in a lecture on luminescence at the Royal Institution.

We are not offering a complete history of fluorescence or phosphorescence, nor explanations of these fascinating

properties; but hints are given below which may serve to assist their elucidation.

M. M. Mascart and Chaumet examined a number of gems under the influence of violet light. One of us (Kunz), in 1889, with Mascart, and with Hallock in 1894, submitted bluish-white, opalescent diamonds (from Bagagen Mines, Brazil,—tiffanyite) to electric light passing through glass of different colours.

Although, as will be mentioned, such compounds as alumina, alkaline earth sulphates, and certain rare earth oxides have been examined in vacuum tubes under the influence of electric sparks, we are not aware of any very extended examination of mineral or chemical substances when subjected to ultra-violet light produced by sparking. E. Becquerel in his "*La Lumière ses Causes et ses Effects*" states that "the electric spark acts only by its light, but its action is more energetic than that of the sunlight by reason of its great intensity and the proximity of the source." Wiedemann and G. C. Schmidt, in an article on luminescence, reached the conclusion that the violet light alone of the electrical discharge does not cause phosphorescence, but is "due to peculiar discharge rays analogous to cathode rays." M. W. Hoffmann confirmed the above. J. Trowbridge reported "the action of the X-rays on this mineral (fluorite) was exactly similar to that of electrification," and concluded, "by them (X-rays) an electrical charge is communicated to fluorescent and phosphorescent substances."

While there may be no question as to the statements above for the particular materials examined—and they are doubtless true for many other substances—yet below may be found observations which do not admit of the sweeping conclusion that all fluorescent and phosphorescent phenomena observed in minerals subjected to rays coming from the sparking of high voltage currents with iron terminals are due to electrification.

The ultra-violet light was produced by a triple spark through quadruple iron terminals (we may so designate them) with a high voltage current. The direct current was taken from a 110 volt circuit passed through a Ruhmkorff coil with a 12-inch spark and stepped up by two Leyden jars in series. The spark was provided with a quartz window surrounded by vulcanite and otherwise insulated to permit comfortable handling. As the number of observations to be made was very great, it was impracticable to remove each individual specimen from the exhibition cases and storage cupboards to the dark room, although this was done in many experiments. Flexible cables 200 feet long were joined to the apparatus. This was placed upon rollers, and could be moved easily to the various aisles between the cases. The Piffard lamp was joined up with the apparatus by insulated wires, further protected by rubber tubing 36 feet long. About 13,000 minerals were thus examined by night.

A large mass of original material has been gathered, of which only a few general observations and tentative conclusions are presented.

The two most responsive minerals to all three forms of activity were found to be willemite and certain diamonds.

1. It was found, as doubtless observed by others, that willemite from Franklin, N.J., is both fluorescent and phosphorescent with the Röntgen rays, ultra-violet rays and when exposed to radium emanations. These properties were retained, although in some instances the specimens were considerably altered by decomposition. Foreign specimens of the same species were not affected at all. The willemite retained its luminescence for more than twenty-four hours after it had been exposed to radium; the latter not being then within 100 feet of it.

2. The calcite from Franklin, N.J., showed a distinct red glow with the ultra-violet rays. This mineral, as well as the willemite, showed very marked peculiarities of colour; the willemite green and yellow-green, the calcite a red glow. These effects were so characteristic that it required but a moment to identify the specimens encountered



| RADIO-ACTIVE MINERALS.              |              | Kunz-Baskerville observations on phosphorescence or fluorescence with— |               |
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| Pitchblende from Joachimsthal .. .. | 7.0          | —                                                                      | —             |
| Pitchblende from Przibram .. .. .   | 6.5          | —                                                                      | —             |
| Pitchblende from Cornwallis .. .. . | 1.6          | —                                                                      | —             |
| Cleveite .. .. .                    | 1.4          | —                                                                      | —             |
| Autunite .. .. .                    | 2.7          | +                                                                      | +             |
| Spyllite .. .. .                    | —            | —                                                                      | —             |
| Various thorites .. .. .            | 0.1          | —                                                                      | —             |
|                                     | 0.3          | —                                                                      | —             |
|                                     | 0.7          | —                                                                      | —             |
|                                     | 1.3          | —                                                                      | —             |
| Orangite .. .. .                    | 2.0          | —                                                                      | —             |
| Monazite .. .. .                    | 0.5          | —                                                                      | —             |
| Xenotime .. .. .                    | 0.03         | —                                                                      | —             |
| Rechnyite .. .. .                   | 0.7          | —                                                                      | —             |
| Fergusonite (two samples) .. .. .   | 0.4          | —                                                                      | —             |
|                                     | 0.1          | —                                                                      | —             |
| Samarakite .. .. .                  | 1.1          | —                                                                      | —             |
| Niobite (two samples) .. .. .       | 0.1          | —                                                                      | —             |
|                                     | 0.3          | —                                                                      | —             |
|                                     | 0.02         | —                                                                      | —             |
| Tantalite .. .. .                   | 6.2          | —                                                                      | —             |
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|                                     | —            | —                                                                      | —             |
|                                     | —            | —                                                                      | —             |
| Monazite sand .. .. .               | —            | —                                                                      | —             |
| Monazite .. .. .                    | —            | —                                                                      | —             |
| Polycrase .. .. .                   | —            | —                                                                      | —             |
| Euxenite .. .. .                    | —            | —                                                                      | —             |
|                                     |              | Amherst County, Virginia .. .. .                                       | —             |
|                                     |              | Barkevik, Norway .. .. .                                               | —             |
|                                     |              | (Auerlite), Green River, North Carolina                                | —             |
|                                     |              | Arendal, Norway .. .. .                                                | —             |
|                                     |              | Cheyenne Cañon, Colorado .. .. .                                       | —             |
|                                     |              | Alexander County, North Carolina ..                                    | —             |
|                                     |              | Hitteroe, Norway .. .. .                                               | —             |
|                                     |              | Llano County, Texas .. .. .                                            | —             |
|                                     |              | Ytterby, Sweden .. .. .                                                | —             |
|                                     |              | Berthier County, Quebec .. .. .                                        | —             |
|                                     |              | Portland, Connecticut .. .. .                                          | —             |
|                                     |              | Arendal, Norway .. .. .                                                | —             |
|                                     |              | Zlatoust, Ural .. .. .                                                 | —             |
|                                     |              | 171st Street and Washington Avenue,<br>New York City .. .. .           | —             |
|                                     |              | Amelia Court House, Virginia .. ..                                     | —             |
|                                     |              | Alexander County, North Carolina ..                                    | —             |
|                                     |              | Rio, Brazil .. .. .                                                    | —             |
|                                     |              | Tvedestrand, Norway .. .. .                                            | —             |
|                                     |              | Near Marietta, South Carolina .. ..                                    | —             |
|                                     |              | Mitchell County, North Carolina ..                                     | —             |

Column 1 is Mdme. Curie's list of rare minerals; 2 is Kunz and Baskerville's ultra-violet ray; 3, Röntgen rays.  
With one exception, neither of the latter show any action.

invarious parts of the collection, without seeing the label, as being from Franklin, N. J.

3. The gangue of all minerals from Pajsberg and Langban, Sweden, also showed this peculiar red glow; the limestone strikingly like the calcite from Franklin, N. J.

4. All the minerals from Mono Lake, California, the colemanite, hanksite, glauberite, iddingsite with ultra-violet rays. The briefest exposure causes them to glow and to retain this luminescence for a considerable time; but none of these minerals either phosphoresced or fluoresced with radium; those tested with magnesium light phosphoresced.

5. Hydro-zincite, from Algiers, showed a remarkable fluorescence, bluish in colour, different from anything in the collection.

6. Autunite and another uranium mineral, from Mitchell County, N. C., fluoresced wonderfully, while foreign specimens of the same species did not. Autunite appears to have two minerals present with it, one an orange and the other a lemon-yellow; one pulverulent and the other in slight tabular crystals. The striking fluorescence obtained with ultra-violet rays was not produced when glass, which is opaque in these rays, was interposed.

The yellow mineral labelled greenockite, from Franklin, N. J., fluoresced in an identical manner, leading one to infer that this is itself a uranium mineral, or else contains the same substance that causes the autunites to fluoresce.

A comparative table is appended. It is self explanatory, and serves to illustrate the conclusions apparently inevitable, namely, the presence of something not previously recognised.

7. Hyalite (botryoidal), on a trachytic rock, from San

Luis Potosi, Mexico, as colourless as the purest water, fluoresced most intensely with a rich British green colour, but these specimens did not phosphoresce. This green fluorescence could be observed when the source of the ultra-violet was five or six feet away. It did not persist on the removal of the source, but flashed in when the rays played upon it. The sparker held near, but without the rays playing upon the specimens, gave no fluorescent effects. Therefore no other conclusion is tenable than that it is the ultra-violet rays that produce this change. The same remark may be made for many of the experiments carried on with the sparker as the source of the ultra-violet light in the examination of these mineral substances. On the other hand, this hyalite neither fluoresced nor phosphoresced when exposed to the magnesium light, Röntgen rays, or radium. In this respect it behaves like the minerals from Mono Lake. No hyalite from other localities responded to any of these activities.

8. By the action of ultra-violet light rays a number of fluorites both phosphoresced and fluoresced; some phosphoresced, and did not fluoresce; some fluoresced, and did not phosphoresce; and some did neither. Further, their colour had apparently no influence in determining this result. It cannot be the fluorine or alkaline earth present that account for this variation. It is more probably the presence of rare earths, such as yttrium and ytterbium. (See CHEMICAL NEWS, vol. lxxxviii., p. 263).

9. Chlorophane has long been known as a mineral very easily rendered luminous by heat. By some authorities it was stated, a century ago, that it was almost always luminous. This variety of fluorspar is found with other fluorspars—sometimes as a vein of purple between veins of

green fluor spar. It proved very responsive to the ultra-violet rays. A variety from Amelia Court House, Virginia, became suddenly luminous from the heat of the hand. This luminosity was lost upon further heating (about red heat), but the phosphorescent properties were restored in a measure by exposure to the Röntgen rays. Trowbridge has observed this with other fluorites (see above). The exquisite coloured fluorescent properties were not regenerated, however. Chlorophane is pyroelectric by attrition, and this peculiarity distinguished it from the ordinary fluorites.

10. It was noted that gypsum from Sicily, when submitted to ultra-violet light, was from two to five times as responsive as specimens from Bavaria and other localities.

11. It was found that those topazes which had lost the sherry colour—the tint so fleeting that some of the museums have been led to protect them from the light—showed no distinct phosphorescence with ultra-violet rays, while the unfaded crystals of that colour responded, but no others.

12. Wernerite from New York phosphoresced, while specimens from foreign sources did not. Many apophyllites and calamines gave no response whatever.

13. Pectolite proved an exceptionally interesting mineral. Every specimen that was exposed to the ultra-violet rays showed an active response; even some that were almost entirely altered to steatite. This is especially striking as some of the specimens from New Jersey were loose delicate aggregates of needle-like crystals. Some were made up of crystals with a texture like felt, others of coarse crystals, and, lastly, the pectolite without any crystalline structure, homogeneous, and one time mistaken for jade, from Tehama County, California.

14. Wollastonite, whether from northern New York, or associated with the rosolite garnet from Mexico, phosphoresced markedly and with some duration, with ultra-violet rays, and responded strongly to radium (300,000).

15. Kunzite, the new variety of spodumene from Pala, California, when exposed to the action of radium of 300,000 activity for a few minutes, became wonderfully phosphorescent, the glow continuing persistently after the removal of the source of excitation. Six hundred grms. of kunzite crystals were excited with 125 m.grms. of radium bromide. Sir William Crookes, in a personal letter, having repeated the experiment, remarks:—"I think this lilac variety of spodumene runs the diamond very close, if it does not surpass it sometimes." Ultra-violet rays caused kunzite to phosphoresce for more than a minute. This remark applies also to the faded or colourless kind. All forms of kunzite become strongly phosphorescent with the Röntgen rays. So pronounced is this that a large crystal excited for five minutes afterwards affected the film of a sensitive photographic plate. And a thirty second exposure caused those gems to glow first golden pink, then white for ten minutes, twenty times the duration of exposure to the X-ray, the glow penetrating two thicknesses of white paper. It is also pyroelectric, assuming a static charge, similar to topaz, when rubbed with a woollen cloth. It does not phosphoresce when heated.

16. The action of the quartz group was interesting. As a rule, quartz proper neither phosphoresced nor fluoresced with ultra-violet rays, allowing them to traverse it without any effect. Hence, the very few exceptions noted were doubtless due to the inclusion of intermixture of other substances. This was apparent in one or two cases of quartz pseudomorphs after barite and fluorite, which phosphoresced evidently from the presence of some remainder of that mineral.

Chalcedonic quartz was also very unresponsive; one example only, from Uruguay, S.A., showing a bluish milky phosphorescence, and a specimen of agate, in which one layer responded between others that did not.

Opal, on the other hand, is frequently phosphorescent, very rarely fluorescent, and sometimes without any action. The variety quincite phosphoresced intensely, as did also

specimens apparently pseudomorphous after gaylussite, which exhibited strong and long continued phosphorescence.

17. Among carbonates, calcite, witherite, strontianite, and barytocalcite, all phosphoresce with ultra-violet light; while arragonite, with occasional exceptions, is very marked in its action, far surpassing calcite. On the other hand, cerussite does not phosphoresce, save in a single specimen from Phoenixville, Pa.

There is here seen again a peculiar phenomenon in minerals from Langban locality, and the suggestion is evident of the existence there, and at points where similar exceptional results appear, as in Mono Lake, of the presence of some rare element (perhaps new) widely diffused in very minute quantities.

A similar indication is given by the behaviour of the glauconite; those from Borax Lake, California, phosphoresce, as do those from Laramie, and from Spain; while Chilean specimens do not.

18. It is notable that tourmaline, which is so markedly pyroelectric, gives no response to ultra-violet light; nor does beryl, save in three specimens from Haddam Neck, Conn.

19. American sapphires of various kinds, spinel, chrysoberyl, and almost all jades, declined to show any effects from ultra-violet rays. Many of the gem minerals, except diamond, opal, and kunzite, are little acted upon.

20. Only two of the rare earth oxides artificially prepared, responded at all to the action of ultra-violet rays, namely, zirconium and thorium dioxides, which phosphoresced strongly. The thorium dioxide remained luminous in the dark for a greater length of time. The zirconium dioxide showed no radio-activity when tested by the electrical and photographic methods. It is strange that two earths forming dioxide are the only ones to exhibit this property. The following oxides were examined:—Yttrium, ytterbium, erbium, gadolinium, samarium, lanthanum, cerium, neodidymium, praseodidymium, thorium, zirconium, titanium, uranium, and variable mixtures of the same. They will be investigated further by one of us (Baskerville).

In view of the fact that these two earths give this characteristic response to ultra-violet light, it became immediately of interest to learn the effect of the light upon minerals carrying those substances in different proportions. The following selected minerals were again subjected to the action of ultra-violet light without a single one of them giving either fluorescence or phosphorescence:—

*Samarskite*—Berthier County, Quebec.

*Thorite* (Orangite)—Arendal, Norway.

" —Barkevik, Norway.

" (Auerlite)—Green River, North Carolina.

*Sipylite*—Amherst County, Virginia.

*Columbite*—Portland, Connecticut.

*Monazite*—Arendal, Norway.

" Zlatoust, Ural.

" 171st Street and Washington Avenue, New York.

" Amelia Court House, Virginia.

" Alexander County, North Carolina.

*Monazite Sand*—Rio, Brazil.

*Monazite*—Tvedestrand, Norway.

*Xenotime*—Cheyenne Cañon, Colorado.

" Alexander County, North Carolina.

" Hitteroe, Norway.

*Euxenite* (in *Samarskite*)—Mitchell County, North Carolina.

*Æschynite*—Hitteroe, Norway.

*Polycrase*—Near Marietta, South Carolina.

*Fergusonite*—Llano County, Texas.

" Ytterby, Sweden.

#### Tentative Conclusions.

1. It seems as though in willemite, hydrozincite, and in the artificial phosphorescent zinc sulphide, zinc oxide, and kunzite, there is present, with the zinc, some element probably not yet determined, that possesses peculiar pro-

erties; or one that in combination with a zinc mineral gives the high luminosity by the application of radium, with the ultra-violet rays, or the Röntgen rays, or other radio-active bodies; an element possibly accompanying zinc and possessing an affinity for it, as polonium has for bismuth, perhaps Phipson's actinium, adverted to.

2. It seems likely also that there exists in fluor spar either yttrium or ytterbium or some other related rare earth, or perhaps several of them, from the variable action of this mineral with the various kinds of rays.

3. When we consider the diverse composition of the numerous minerals coming from Borax Lake, and that it consists of the evaporated remains of an inland sea, there seems to be present some element which is very highly active, but is not responsive to radium, and which appears in every single mineral found here. This is evidently a substance not necessarily radio-active itself, but one that may possess the same or allied properties with the substance found with the zinc-minerals.

4. The substance present in calcite, from Franklin, N. J., and from Langban and Pajsberg, Sweden, is probably yet another body, as it does not respond to radium; although the willemite found with it at Franklin becomes luminous at the approach of radium or ultra-violet light, as if they were fairy wands.

5. There probably exists in autunite, and another yellow-brown uranium mineral from Texas, a fluorescent substance which differs from anything we have noted in the study of the minerals of the collection.

6. In the hyalite from San Luis Potosi, a volcanic mineral, there is present something that responds with a beauty of colour that strikingly reminds one of nitrate of uranium; this is probably a physical change, yet may prove to be still another substance.

7. The most responsive of all, however, were the diamonds containing that peculiar substance that gives them what is known as the blue-white colour—fluorescent like anthracene, and holding the luminosity for a long time—to which one of us (K.) gave the name of Tiffanyite in 1895.

In the examination of more than 15,000 diamonds from British Guiana and elsewhere, 44 were selected. After an exposure of 60 seconds to ultra-violet rays, these 44 diamonds phosphoresced brilliantly and continued to glow for a long time after exposure. The luminosity was so great that it penetrated one thickness of white velvet and from nine to twelve thicknesses of tissue and blue linen paper. But they did not exhibit their light through black velvet, nor apparently were they affected by the ultra-violet rays when surrounded by black velvet. These diamonds when glowing brilliantly showed absolutely no action upon the barium platino-cyanide screen, nor upon screens of phosphorescent zinc sulphide or calcium sulphide.

The most remarkable specimen was a diamond of 15½ carats. (This was exhibited). This stone possesses the power of absorbing sunlight and emitting it in the dark. An arc lamp will cause it to store up energy and to give it out as light in the dark. Even a small hand-lamp of one candle power has caused this diamond to phosphoresce. It responds to polonium, to the Röntgen rays, and to the ultra-violet rays; to the rays that pass through a violet glass, and to radium, even in a more marked degree than willemite.

The print shown was made from a negative obtained by exposing a sensitive photographic plate to the 15 carat blue-white and a 15 carat transparent black stone, thin white paper intervening, after they had been exposed to ultra-violet light for one minute. The print is the result; except that the print of the black stone has been coloured to show the reddish phosphorescence given out by it.

After another exposure of one minute, to our surprise the black stone glowed red for fifteen minutes, almost surpassing the white phosphorescence of the blue-white stone. At the end of fifteen minutes the red glow subsided, while

the white stone phosphoresced five minutes longer; the light being held twenty minutes after exposure.

As stated above, from the work of Wiedemann and Schmidt, Hoffmann and Trowbridge, it appears that the phosphorescent and fluorescent effects observed by the action of ultra-violet light produced by sparking with such metals as iron, is not due to this cause at all, but may be accounted for by the accumulation of an electric charge. The diamonds were "grounded" by placing directly upon the iron radiator in the room and similar observations made, as when the precious stones were insulated.

It is interesting here to note that Marckwald reported the property of phosphorescence with polonium as belonging only to Brazilian diamonds. Rosenheim found that the rays from radio-active polonium possess the property of inducing fluorescence in a number of diamonds from different localities. The rays emitted by the diamonds under these conditions affect the retina and photographic plate. "This actinic activity of the diamond," he says, "like its visible fluorescence, is entirely dependent on the presence of the polonium, not persisting after the removal of the latter. Even after long exposure to polonium rays no induced radio-activity could be detected."

Almost all diamonds of various weights and from many localities and different colours, fluoresce and phosphoresce more or less with radium, except the black or carbonado. The degree to which these phenomena are observed is no criterion of the grade of the gem, however, as stones with flaws often fluoresce with even greater brilliancy than the pure jewels.

8. It is quite evident through our study of the collection that one or the other of these forms of luminosity and activity may have a value to detect elements or compounds that have escaped notice or are present in the minerals as impurities. These forms of investigation may also prove of value in chemical analyses. There should be a use for this line of research also in petrological determinations, as the slightest phosphorescence or fluorescence would aid in determining and locating a mineral, no matter how minute in quantity. This we have done in several instances.

The original ultra-violet lamp was that of Gori, of Munich, altered by the English into the St. Bartholomew lamp, and again improved and made practicable in the United States under the name of the Piffard lamp, after Dr. H. G. Piffard, of New York, who has used it with much success in medical practice. It is an instrument of great utility, and, in the convenient form with which we worked, cannot fail to prove a valuable mineralogical and chemical as well as medical adjunct. The lamp will be useful in many instances for mineralogical determination, at times to detect impurities which have escaped analysts and others.

9. In all observations on the effect of radium, ultra-violet light, and the X-rays to determine whether an object becomes fluorescent or phosphorescent under the influence of either, it is essential that the eyes become thoroughly accustomed to the change of conditions when one is in a dark room.

This usually requires from ten to twenty minutes, and in some cases half-an-hour. Attention to this has been called by preceding observers. Whether it be due to the accumulation of the visual purple, which von Kries states is a substance that supplies the retinal basis of vision at low luminosities, and whose accumulation is accountable for the great increase in sensitiveness of the dark adapted eye, or to the ordinary physical changes in the optical lenses, or both in part, we do not undertake to decide. But it was learned that it was advisable just before the source of excitation was removed from the material examined that the eyes be closed and opened again after the removal. Else, as was noted, the residual flash that remained may be mistaken for phosphorescence. In most of the experiments carried out three observers watched each test. When there was the least disagreement, the tests were

repeated a sufficient number of times until a unanimous agreement was arrived at.

10. The Röntgen rays have been used with great success to locate fractures, mis-growths, deformities, and abscesses in the bony processes; but as far as we are aware, little success has attended efforts to locate ruptures, growths, or peculiarities of the veins, intestines, &c., by this means. There seems a possibility, however, that if a highly fluorescent or phosphorescent substance could be injected into the veins, the stomach, or the intestines, it would be feasible to locate lesions, growths, and other peculiarities of these organs; possibly also to locate accretions and kidney or bladder concretions, especially calcareous, as well as, possibly, peculiarities in the structure of the heart and other organs. This it might be practicable to do by means of inert but phosphorescing materials in solution given in the food, or injected into the stomach or intestines when they are quite empty. It might be that a nearer location could be effected in the organs desired to be examined if impalpable powders be given with the food. If it were possible to inject such a substance into the blood, the entire vein structure of the body might be rendered visible as well as the bony part. It seems not unlikely that such an active agent as radium, or ultra-violet light, may yet be found a great accessory in diagnosis and autopsies, as they have given promise of marvellous curative values in certain diseases. After presenting this paper we were informed that Dr. Morton and Mr. W. J. Hammer have investigations along these lines now in progress. The latter has prepared a highly interesting lecture on radium.

11. The final part of the work planned was an investigation of the influence of cathode rays upon gems and the gem material of these collections. The method utilised in the classical investigations of Becquerel, Crookes, and de Boisbaudran on the fluorescence and phosphorescence of a number of substances, especially alumina and the rare earths, *in vacuo*, and spectroscopic examination of the light emitted therefrom, offer possibly an answer to questions as to the nature of such substances as give tiffanyite its unique properties, for example. Small amounts may be used; the destruction of such valuable gems in chemical analysis being out of the question. The time at our disposal having been utilised in securing the observations briefly outlined above, we were forced to discontinue the research for a time to work up a great quantity of experiments made of which these are only a few, although a number of Crookes tubes have been charged with material and exhausted. We hope to complete the phase of the undertaking, but confess, from what has been indicated above, that things have been seen that shine like a "pillar of fire by night," and beckon us on.

12. From the summarised observations on minerals recited above, it appears that there are evidently two properties recognisable—radio-activity and a property that responds to this activity. It is hence seen that we have two classes of bodies—radio-active and those that are affected by radio-activity; and that these groups may be again divided into several minor divisions:—

1. Those that respond to the radio-active bodies.
2. Those that do not respond.
3. Those that respond to ultra-violet rays.
4. Those that respond to Röntgen rays.
5. And then again, some will not respond to either one, two, or all of the three forms.

We seem to find here an analogy to certain well-known facts in electricity and magnetism; some bodies that are active, and others that are acted upon in several different forms, which are evidently closely related, and yet are distinct in their modes of action. We are privileged, therefore, to offer for mineral substances a—

#### *Tentative Classification.*

Those minerals:—

1. Not responding to radium, ultra-violet, or Röntgen rays.
2. Responding to radium only.

3. Responding to ultra-violet rays.
4. Responding to Röntgen rays.
5. Responding to radium and ultra-violet rays (not to Röntgen rays).
6. Responding to radium and Röntgen rays (not to ultra-violet rays).
7. Responding to ultra-violet and Röntgen rays (not to radium).
8. Responding to radium, ultra-violet rays, and Röntgen rays.

As for mineralogical determination, no large apparatus is necessary as is used in medicine or physiological investigations. In fact very simple apparatus is sufficient. Therefore we are devising a series of appliances so that the entire apparatus may probably be purchased for much less than one hundred dollars.

We are also preparing a list of minerals, selecting those most readily obtainable, to illustrate these phases of activity or inactivity with the three useful accessories. For a small expenditure any school or college can obtain them for comparative study.

As the electric furnace has given us carborundum, artificial graphite, and a series of absolutely new carbides, because with it we have attained temperatures of a height unknown before its introduction—as the production of low temperatures has resulted in the liquefaction of all known gases and assisted in the discovery of new ones—so perhaps the application of these forms of energy may give us the means of identifying substances that all our earlier methods of observation have escaped; and it may be that we shall have a new series of elements. We are clearly on the threshold of a new field of scientific facts, and perhaps generalisation and laws, which may yield results in the twentieth century as interesting and remarkable as the electrical discoveries were in the nineteenth. Indeed, some have already discarded atomic chemistry and assumed ionic chemistry, while pioneers like Crookes, J. J. Thomson, and Lodge vouchsafe "protyle," "corpuscles," and "electrons," with more or less experimental verification, although they do not quite reach Ostwald's metaphysical view.

Here we have to mention the aid given us in having access to the collections, the construction of a special dark room, every facility of the Museum's workshops, the encouragement and advice given by the Museum's able director, Dr. H. C. Bumpus, and to the attention and assistance of Dr. L. P. Gratacap, and Mr. L. P. Seymour, and Mr. Smith, of the Mineralogical Department, and Mr. Dahlgren, the Museum photographer.

### POTABLE WATERS IN SOUTH-WEST LANCASHIRE.\*

ON THE NATURE AND QUALITY OF SOME POTABLE WATERS IN SOUTH-WEST LANCASHIRE.

By Professor J. CAMPBELL BROWN, D.Sc.

THESE waters may be classified under three heads:—

- I. Surface waters.
- II. Deep-well or bore waters.
- III. Shallow-well or spring waters.

I. The earliest of the first class is the Rivington supply, introduced into Liverpool in 1857. Although this water has been in daily use for forty-six years, and has received analysis for sanitary purposes frequently from 1850 down to the present year, no complete analysis of the mineral constituents has been made by anyone, so far as I have been able to ascertain. I therefore made a full analysis a short time ago for the purposes of this paper. It is interesting

\* A Paper read before the British Association, Southport Meeting, 1903.

|                                                    | 3.                                            | 4.    | 5.         | 6.                                | 7.          | 8.      | 9.                                        | 10.     | 11.          | 12.                                     | 13.   |
|----------------------------------------------------|-----------------------------------------------|-------|------------|-----------------------------------|-------------|---------|-------------------------------------------|---------|--------------|-----------------------------------------|-------|
|                                                    | Erismagh.                                     |       | Spade Min. | Dilworth.                         | Tilled and. | Quarry. | Sandstone above a colliery.               | Quarry. | Netherby Wd. |                                         |       |
|                                                    | 1892.                                         | 1900. | 1892.      | 1892.                             |             |         |                                           |         | 1892.        | 1899.                                   | 1903. |
| Total solids                                       | 7.8                                           | 8.64  | 8.76       | 10.92                             | 28.74       | 24.8    | 40.8                                      | 18.56   | 14.34        | 16.6                                    | 15.2  |
| Organic carbon                                     | —                                             | —     | —          | —                                 | —           | —       | 0.064                                     | —       | —            | 0.049                                   | —     |
| Organic nitrogen                                   | —                                             | —     | —          | —                                 | —           | —       | 0.023                                     | —       | —            | 0.021                                   | —     |
| Oxygen consumed in fifteen minutes                 | —                                             | 0.084 | —          | —                                 | —           | —       | —                                         | 0.000   | —            | 0.002                                   | 0.001 |
| Oxygen consumed in three hours                     | —                                             | 0.163 | —          | —                                 | —           | —       | —                                         | 0.012   | —            | 0.012                                   | 0.002 |
| Ammonia                                            | 0.011                                         | 0.002 | 0.006      | 0.011                             | 0.002       | 0.000   | 0.002                                     | 0.002   | 0.000        | 0.002                                   | 0.003 |
| Ammonia by distillation with alkaline permanganate | 0.021                                         | 0.01  | 0.012      | 0.025                             | 0.002       | 0.010   | 0.000                                     | 0.003   | 0.002        | 0.000                                   | 0.002 |
| Nitrogen as nitrates                               | 0.021                                         | 0.000 | 0.021      | 0.000                             | 0.000       | 0.24    | 0.000                                     | 0.35    | 0.175        | 0.241                                   | 0.3   |
| Combined chlorine                                  | 1.1                                           | 1.1   | 1.4        | 1.3                               | 2.2         | 2.3     | 1.8                                       | 3.8     | 2.3          | 2.2                                     | 2.23  |
| Temporary hardness                                 | —                                             | —     | —          | —                                 | —           | —       | 27.5°                                     | —       | 2.43°        | 0.72°                                   | 3.27° |
| Permanent hardness                                 | —                                             | —     | —          | —                                 | —           | —       | 7.5°                                      | —       | 7.57°        | 6.71°                                   | 6.73° |
| Total hardness                                     | 3.64°                                         | 3.9°  | 3.77°      | 6.29°                             | —           | —       | 35°                                       | 10°     | 10°          | 7.43°                                   | 10°   |
|                                                    | Neutral; contain iron, but no iron organisms. |       |            | Alkaline; contains trace of iron. |             |         | Alkaline; contain iron, but no organisms. |         |              | No organisms of any kind in this water. |       |

|                                            | 14.           | 15.   | 16.    | 17.                 | 18.               | 19.               | 20.                   | 21.    | 22.       | 23.                | 24.                                 |
|--------------------------------------------|---------------|-------|--------|---------------------|-------------------|-------------------|-----------------------|--------|-----------|--------------------|-------------------------------------|
|                                            | Stock's Well. |       |        | The same deep bore. |                   |                   | St. Helen's District. |        |           | Ormskirk District. |                                     |
|                                            |               |       |        | Depth,<br>60 ft.    | Depth,<br>120 ft. | Depth,<br>500 ft. |                       |        |           |                    |                                     |
|                                            | 1892.         | 1899. | 1903.  |                     |                   |                   |                       |        |           |                    |                                     |
| Total solids .. .. .                       | 15.2          | 18.6  | 14.4   | 18.6                | 17.2              | 27.6              | 51.3                  | 25.2   | 33.62     | 76                 | 16.24                               |
| Organic carbon .. .. .                     | —             | 0.029 | —      | —                   | —                 | —                 | —                     | —      | 0.211     | —                  | —                                   |
| Organic nitrogen .. .. .                   | —             | 0.011 | —      | —                   | —                 | —                 | —                     | —      | 0.031     | —                  | —                                   |
| Oxygen consumed in fifteen minutes .. .. . | —             | 0.001 | 0.004  | 0.004               | —                 | 0.000             | —                     | —      | —         | 0.000              | 0.000                               |
| Oxygen consumed in three hours .. .. .     | —             | 0.005 | 0.006  | 0.006               | —                 | 0.004             | —                     | —      | —         | 0.021              | 0.000                               |
| Ammonia .. .. .                            | 0.002         | 0.000 | 0.001  | 0.004               | 0.005             | 0.002             | 0.002                 | 0.000  | 0.000     | 0.002              | 0.000                               |
| Ammonia by distillation .. .. .            | 0.005         | 0.000 | 0.001  | 0.004               | 0.005             | 0.002             | 0.000                 | 0.002  | 0.002     | 0.006              | 0.000                               |
| Nitrogen as nitrates .. .. .               | 0.196         | 0.24  | 0.22   | 0.371               | 0.35              | 0.173             | 0.131                 | 0.482  | 0.415     | 3.31               | 0.59                                |
| Combined chlorine .. .. .                  | 1.9           | 2.    | 1.76   | 2                   | 1.8               | 4.25              | 4.6                   | 2.5    | 3.1       | 8.75               | 2.7                                 |
| Temporary hardness .. .. .                 | 2.86°         | —     | 3.11°  | —                   | —                 | 2.56°             | 21.25°                | 7.75°  | —         | —                  | —                                   |
| Permanent hardness .. .. .                 | 6.57°         | 9.14° | 7.14°  | —                   | —                 | 6°                | 13°                   | 10.5°  | —         | —                  | —                                   |
| Total hardness .. .. .                     | 9.43°         | 9.14° | 10.25° | —                   | —                 | 8.56°             | 34.25°                | 18.25° | 20°       | —                  | —                                   |
| No organisms of any kind in this water.    |               |       |        | Neutral.            |                   |                   | Alkaline.             |        | Alkaline. |                    | Excessively hard; no carbonic acid. |
|                                            |               |       |        |                     |                   |                   |                       |        |           |                    |                                     |

to compare the figures with those of an analysis of the Vyrnwy supply made for the Corporation of Liverpool in 1898.

*Analyses Expressed in Parts per 100,000.*

|                                                                        | 1.<br>Rivington water.<br>Clear, but not<br>filtered.<br>1903. | 2.<br>Vyrnwy water.<br>Strained, but<br>not filtered.<br>1898. |
|------------------------------------------------------------------------|----------------------------------------------------------------|----------------------------------------------------------------|
| Total solids in solution..                                             | 10'16                                                          | 3'98                                                           |
| Loss on ignition .. ..                                                 | 2'74                                                           | 1'05                                                           |
| Alkalis .. .. .                                                        | 1'06                                                           | 0'518                                                          |
| Magnesia.. .. .                                                        | 0'461                                                          | 0'127                                                          |
| Lime .. .. .                                                           | 1'735                                                          | 0'706                                                          |
| Oxide of iron .. ..                                                    | 0'016                                                          | 0'099                                                          |
| Oxide of manganese ..                                                  | 0'017                                                          | 0'059                                                          |
| Chlorine .. .. .                                                       | 1'500                                                          | 0'6                                                            |
| SO <sub>3</sub> .. .. .                                                | 2'144                                                          | 0'6                                                            |
| Silica .. .. .                                                         | 0'463                                                          | 0'214                                                          |
| Total mineral matter ..                                                | 7'417                                                          | 2'923                                                          |
| Combined CO <sub>2</sub> .. ..                                         | none                                                           | none                                                           |
| Organic carbon .. ..                                                   | 0'343                                                          | 0'498                                                          |
| Organic carbon after filtra-<br>tion .. .. .                           | 0'323                                                          | 0'331                                                          |
| Organic nitrogen .. ..                                                 | 0'064                                                          | 0'064                                                          |
| Organic nitrogen after<br>filtration .. .. .                           | 0'052                                                          | 0'042                                                          |
| Nitrogen as nitrates ..                                                | trace                                                          | 0'109                                                          |
| Ammonia .. .. .                                                        | 0'003                                                          | 0'006                                                          |
| Ammonia from distilla-<br>tion with alkaline per-<br>manganate .. .. . | 0'009                                                          | 0'017                                                          |
| Oxygen required to oxidise<br>in fifteen minutes ..                    | 0'048                                                          | 0'048                                                          |
| Oxygen required to oxidise<br>in three hours .. ..                     | 0'084                                                          | 0'296                                                          |
| Acidity due to organic<br>matter equivalent to<br>CaO .. .. .          | 0'19                                                           | 0'2                                                            |
| Dissolved gases per litre :—                                           |                                                                |                                                                |
| Oxygen .. .. .                                                         | —                                                              | 5'45 c.c.                                                      |
| Nitrogen .. .. .                                                       | —                                                              | 11'53 "                                                        |
| CO <sub>2</sub> .. .. .                                                | —                                                              | none                                                           |

The most important practical difference is not very evident on the face of the analytical figures, and I may as well point it out. It is the much smaller quantity of organic compounds of iron in the Rivington water. The difference in the iron is as 0'016 in Rivington to 0'099 in Vyrnwy.

There is no means of exactly determining quantities of organic matter itself. Perhaps the best figure showing the difference to which I allude is that for oxygen consumed in three hours—namely, the ratio of 0'084 in Rivington to 0'296 in Vyrnwy.

The practical difficulties due to this difference are :—

1. Filtration and decolorisation.
2. Action on lead.
3. Obstruction of the pipes by deposits and growths.

The Rivington water is slightly coloured and contains a small quantity of organic compounds of iron, to which colour is generally due. Although the two waters are so much alike, there is none of that development of iron organisms in Rivington water which is so common in Welsh waters like that of the Vyrnwy. It is excellent water for domestic use, and is likely to remain so, now that Liverpool has the power to repress the pollution of the watershed by dwelling-houses, inns, and farms.

Good examples of Lancashire catchment waters are those supplying Preston, Nos. 3 to 6. Occasionally agricultural land yields good water, as in No. 7. But water from agricultural land generally yields water too impure to be fit for domestic use. Intermediate between surface water and well water are waters collected in quarries. Nos. 8, 9, and 10 furnish examples of these.

The head waters of most of the Lancashire rivers are also of good quality; but by the time that they reach south-

west Lancashire they have received so much pollution, both from human dwellings and manufactories, that it is impossible to use them, and they need not be further mentioned here.

**II. Deep-Bore Water.**—The City of Liverpool was, until recently, supplied to a great extent from deep wells and bores in the sandstone. The rock is becoming gradually saturated with the partly oxidised waste water of our sewers and streets, and the wells are, one after another, falling into disuse, only three being still in use for public supply. In smaller places deep wells are still the best kind of supply.

As typical examples of good deep-bore water from the Keuper sandstone of Lancashire, two wells supplying Widnes may be mentioned—Analyses 11 to 16.

It is interesting to compare the composition of the same bore at different depths. Nos. 17 to 19 show the quality of the water at different depths of a deep bore at Newton. This is typical of many bores in Lancashire and Cheshire, which have been examined as the bore was deepened.

Nos. 20, 21, and 22 are deep wells in the St. Helens district.

No. 23 is from a deep well in the Ormskirk district, where much lime and magnesia are found in all the underground waters.

**III. Shallow Wells.**—Shallow wells—that is, wells under 32 ft. in depth—which are still the chief source of supply in country places, vary greatly in composition, and are usually unfit for domestic use. A well must not, however, be assumed to be bad because it is shallow. Those which yield good potable water have generally but small quantities of solids in solution; they are soft, almost free from nitrates, have no nitrites, the figures for ammonia are high, and they consume much oxygen. They are alkaline as a rule, except towards the Yorkshire side, and contain numerous and varied organisms—vegetable, such as diatoms, desmids, confervæ, and bacteria; and animal, such as cyclops, paramécia, and many others.

Exceptions occur in which the total solids are very high; for example, in No. 25 the total solids in solution amounted to 108 parts per 100,000; in No. 26 to 112'5—this was ferruginous, but contained no living organisms; and in No. 27, 175'6. This last was the only potable water available, and therefore it had to be used, although the dissolved salts were so excessive.

**IV. Although the iron organisms which have given much trouble in some Welsh waters have created no similar difficulties in Lancashire, this is not because they are altogether absent, but because they do not flourish and multiply.**

*Iron Organisms in Lancashire Waters.*

Attention was first directed to the occurrence of deposits formed by iron organisms in potable waters in Lancashire in the winter of 1890-91, when decomposed vegetable masses and strings of bacteria—in their earlier forms, nearly colourless, and in their older state strongly coloured by ferric oxide—were observed in water coming from the deep bore of Bootle well belonging to the Corporation of Liverpool.

These ferruginous strings were reported at the time to be "tangled masses, enclosing in their meshes particles of sand and filaments."

These red masses came up with the water of the 1300 ft. deep bore.

They were deposited and grew on the stones lying in the well and headings. They were liable to be drawn up by the pumps and pass into the water supply. They had no effect on health, but were unsightly, and they therefore required investigation and repression.

Under the microscope they resembled the iron organisms known at that time by the name of *Crenothrix Kuhniana*, which had caused a great deal of trouble in the water reservoirs and channels of the waterworks of Rotterdam and Berlin by increasing to such an extent as to interfere with

the flow of water, and which have since been found in other Continental water supplies.

They were traced as far down in the 1300 ft. bore as 700 ft. At that point they were being carried up with the water from a great depth, and were in a living state, capable of being kept alive in the laboratory.

The water of this bore, like that of the Bootle well generally, was well aerated.

They were not formed in the 700 ft. bore adjoining, nor in any of the sixteen other bores which had less depth, although they grew freely on stones in the mixed water from all the bores.

The occurrence of these organisms in the well water was checked by plugging the deep bore. After this they disappeared from the well and did not find their way into the other bores. The large quantity of organic matter in the deep-bore water containing the organisms is noticeable, compared with the quantity after the deep bore was plugged, and has probably afforded food for the iron organisms.

I have shown elsewhere that the multiplication of these organisms is dependent on the presence in the water of an organic compound of iron, having an acid reaction.

The next occasion on which iron organisms were observed was in 1892, in water from agricultural land, which gave a slight deposit of iron oxides, with diatoms, and a few iron organisms. A well near Wigan, and another water near Garstang, in 1894, contained them, although they did not develop to any extent. Neither sample was acid, but contained a little iron and organic matter.

*Composition of Bootle Well Water in 1890-94.*

|                                                  | 1890-91. | After plugging<br>deep bore,<br>February, 1891. | 1894. |
|--------------------------------------------------|----------|-------------------------------------------------|-------|
| Total solids .. .. .                             | 40       | 42·08                                           | 42    |
| Ammonia .. .. .                                  | 0·003    | 0·003                                           | 0·000 |
| Ammonia yielded by alka-<br>line permanganate .. | 0·004    | 0·006                                           | 0·000 |
| Organic carbon .. .. .                           | 0·152    | 0·065                                           | 0·052 |
| Organic nitrogen .. ..                           | 0·034    | 0·011                                           | 0·022 |
| N as nitrates .. .. .                            | 0·401    | 0·459                                           | 0·459 |
| Chlorine .. .. .                                 | 4·2      | 4·1                                             | 4·1   |
| Hardness .. .. .                                 | 25°      | 26°                                             | 26½°  |

No iron; alkaline re-  
action; no organisms.

The only example of water containing bicarbonate of lime which I have met with containing any iron organisms was one in 1895. It was from a shallow well with an iron pump, and they did not develop to any extent.

*Analysis Yielded*

|                               |       |
|-------------------------------|-------|
| Total solids.. .. .           | 74·08 |
| Ammonia .. .. .               | 0·004 |
| Ammonia by permanganate .. .. | 0·008 |
| Nitric nitrogen .. .. .       | 0·371 |
| Combined chlorine .. .. .     | 6     |

No iron in solution, but a slight deposit derived from the pump; there was a white scum formed by evaporation of the carbonic acid; also decayed vegetable matter, and a few iron organisms (cladotrix).

Two examples occurred in 1896 from a spring well after the water, which was neutral, had been conveyed in iron pipes. The water contained organic matter, and the organisms did not develop much in either case.

In 1899 they were found in a tarn in the Lake District which contained 9 parts of dissolved salts, including 0·2 part of dissolved oxide of iron per 100,000. These waters contained also organic matter and were acid, and developed an organism which was at that time called *Cladotrix dichotoma*.

THE SOLUBILITY OF HYDRATED SULPHATE  
OF LIME IN SOLUTIONS OF SEA-SALT.

By ALEX. D'ANSELME.

SATURATED solutions of chloride of sodium (natural brine), which serve as the raw material in the manufacture of soda-ammonia, are obtained by dissolving the solid sea-salt obtained from salt marshes, in fresh water free from salts of magnesium.

These solutions always contain a considerable proportion of sulphate of lime, which is always greater than the proportion of this salt soluble in pure water.

Analyses made regularly for several years, dealing with hundreds of thousands of tons of the dissolved salts, have never given less than 2·5 grms. of  $\text{SO}_4\text{Ca}$  per litre of brine.

This shows that commercial chloride of sodium always contains sulphate of calcium, which confirms the observations of M. Cloez in a recent paper (*Bull. Soc. Chim.*, Series 3, vol. xxix., p. 167). This impurity is of considerable inconvenience in the utilisation of these brines, as it is precipitated under the influence of currents of  $\text{CO}_2 + \text{NH}_3$  gas.

The examination of these solutions, undertaken some months ago, has led me to determine the solubility of sulphate of calcium in solutions of chloride of sodium of different strengths, and at about the ordinary temperatures.

Chemically pure solutions of chloride of sodium, of which the concentration is in simple relation with the normal strength, are brought into contact with an excess of pure sulphate of calcium, and agitated in a continuous and regular manner. For this purpose the flasks are fixed on to a wheel revolved by a dynamo.

The whole is plunged into a thermostat, which assures a constant temperature during the whole of the experiment, at least within a degree.

Under these conditions, I made experiments on the time necessary to reach equilibrium, by taking samples of the liquid every hour from the same flask in the presence of a continual excess of  $\text{SO}_4\text{Ca}$ . Equilibrium was always reached in less than three hours agitation. Nevertheless, my definite final analyses were not made until after nine or ten hours of continuous agitation.

The lime, precipitated by  $\text{CO}_3\text{Am}_2$ , was estimated first in the form of pure  $\text{CaO}$ , then in the form of sulphate, after having been taken up with sulphuric acid.

Two series of experiments were made, one at 14°, the other at 20°.

*Results found.*

| NaCl, per litre. |           | Anhydrous $\text{SO}_4\text{Ca}$ dissolved,<br>per litre. |             |
|------------------|-----------|-----------------------------------------------------------|-------------|
| Grms.            | Molecule. | At 14°.                                                   | At 20°.     |
| 0                | N/∞       | 1·70                                                      | 2·10        |
| 2·925            | N/20      | 2·32                                                      | 2·70        |
| 5·850            | N/10      | 2·79                                                      | 3·15        |
| 11·70            | N/5       | 3·41                                                      | 3·75        |
| 14·62            | N/4       | 3·68                                                      | 4·00        |
| 29·25            | N/2       | 4·40                                                      | 4·70        |
| 58·50            | N         | 5·72                                                      | 6·00        |
| 87·75            | N½        | 6·58                                                      | 6·85        |
| 102·3            | N¼        | 6·90                                                      | 7·15        |
| 117·0            | 2N        | 7·10                                                      | 7·30        |
| 131·6            | 2N½       | 7·20 (max.)                                               | 7·30 (max.) |
| 146·2            | 2N¼       | 7·10                                                      | 7·15        |
| 160·8            | 2N½       | 7·00                                                      | 7·05        |
| 175·6            | 3N        | 6·80                                                      | 6·80        |
| 204·7            | 3N¼       | 6·30                                                      | 6·30        |
| 234·0            | 4N        | 5·90                                                      | 5·90        |
| 263·2            | 4N½       | 5·50                                                      | 5·52        |
| 292·6            | 5N        | 5·30                                                      | 5·30        |

These figures form two regular curves very close together.

We see that :—1. As the proportion of NaCl increases, the solubility increases at first rapidly, then slowly until it reaches a maximum.

2. This maximum corresponds to about 130 grms. per litre (about 2N<sub>1</sub>). It appears to depend to a slight extent on the temperature. The observations were more numerous in the neighbourhood of the maximum so as to determine its position with accuracy.

3. In solutions only slightly charged with NaCl, the solubility varies but little with the temperature.

4. At saturation-point the solubility is decidedly greater than it is in pure water.

The figures given above differ considerably from those given by M. Cloez (*loc. cit.*), but they agree very closely with those given by F. Claméron (*Bull. Soc. Chim.*, vol. xxviii., p. 50), who found a maximum of 7.5 grms. of anhydrous SO<sub>4</sub>Ca at 23°, in solutions containing from 135 to 140 grms. of NaCl per litre.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 9.

## COLORIMETRIC ESTIMATION OF BISMUTH.

By PAUL PLANÈS.

WHEN an acid aqueous solution of a salt of bismuth is treated with an aqueous solution of iodide of potassium, we obtain a brown precipitate of iodide of bismuth.

If, on the other hand, we add the bismuthic solution to the solution of iodide of potassium, at first we do not obtain any precipitate, but only a more or less deeply coloured orange-yellow colouration, and it is only on adding an excess of the bismuthic solution that we observe the formation of a precipitate of BiI<sub>3</sub>.

We have observed that if, when operating as in the first case, we take the precaution of adding its own volume of glycerin at 30° to the bismuthic solution, the subsequent addition of iodide of potassium does not cause any precipitate, but only an orange colouration; the BiI<sub>3</sub> remains in solution owing to the glycerin; in the same way the addition of glycerin at 30° in the second case prevents any precipitation of BiI, no matter what excess of bismuthic solution there may be.

The glycerin plays a double part:—

a. It prevents the precipitation of BiI;

b. When present in sufficient proportion, it prevents the dissociation of the salts of bismuth by dilution with water, which enables us to work with very slightly acid bismuthic solutions, and reduces to a minimum the chances of the dissociation of the iodides by the nitric acid.

We have observed, further, that the orange-yellow colouration obtained under the above optimal conditions was proportional:—

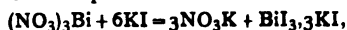
1. To the amount of KI added in excess to the bismuthic solution;

2. To the amount of bismuthic solution added in excess to the solution of KI.

Thus, by the mixture of two colourless solutions, we obtain a perfectly limpid coloured solution, and there is a definite relation between the amounts of the substances mixed and the intensities of the corresponding colourations; these elements are sufficient for a colorimetric estimation.

For the estimation of bismuth in any form, it is necessary to have at our disposal a standard solution of bismuth containing a considerable amount of glycerin, and a titrated solution of potassic iodide, also containing a large amount of glycerin.

To estimate the strength of the standard bismuth solution, we have examined the reaction in which the maximum amount of iodide of potassium should be used—



and according to which, one molecule of neutral nitrate of bismuth corresponds to six molecules of potassic iodide; in other words, 208 grms. of Bi corresponds to 996 grms. of KI; or again, 1 gm. of Bi corresponds to 4.79 grms. of KI.

We adopted therefore a standard solution of bismuth at 1/100th, and a titrated solution of potassic iodide at 5/100ths.

**Preparation of the Standard Solution of Bismuth at 1/100th.**—Our first care was to obtain pure bismuth. For this purpose, following the instructions given by Descaups, we dissolved commercial bismuth in nitric acid, (this transformed the tin present into insoluble metastannic acid); decanted, added an excess of ammonia (this precipitated the oxide of bismuth, and dissolved the Ag<sub>2</sub>O and CuO); treated the oxide of bismuth with a 2 per cent solution of soda (this removed the Pb and As); dissolved 4 parts of the oxide in 1 part of nitric acid, and precipitated with distilled water in the form of basic nitrate of bismuth. The sub-nitrate obtained in this manner, after various corroborative assays to confirm the absence of impurities, (Marsh's apparatus, H<sub>2</sub>SO<sub>4</sub>, NH<sub>3</sub>, &c.), was reduced by charcoal after being thoroughly well mixed, and we used the ingot thus obtained, after having once more verified its purity.

**Method of Procedure.**—Dissolve 1 gm. (accurately weighed) of the pure bismuth in a mixture of 3 c.c. of official nitric acid and 2.8 c.c. of distilled water, following the instructions in the Codex, and after solution make up to 100 c.c. with glycerin at 30°. The standard solution thus obtained does not change.

**Preparation of the Titrated Solution of Iodide of Potassium at 5/100ths.**—Dissolve 3 grms. of pure KI (obtained according to the instructions given in the Codex, but using CO<sub>2</sub>K<sub>2</sub> instead of KOH—the CO<sub>2</sub>K<sub>2</sub> itself should be prepared by the calcination of the purified acid tartrate of potassium) in 5 c.c. of distilled water and make up to 100 c.c. with glycerin at 30°. This should be kept away from the light in a yellow glass bottle.

**Applications.**—As an example, we will take the titration of a commercial sub-nitrate of bismuth.

It is evident that, to facilitate the comparisons, we should try to obtain a solution of the sub-nitrate, containing a proportion of bismuth similar to that present in an equal volume of the standard solution.

Now a normal sub-nitrate of bismuth should contain 76.78 per cent of Bi<sub>2</sub>O<sub>3</sub>; that is to say, 68.83 per cent of pure bismuth; from which it results by a very simple calculation, that to obtain the solution in question we must take 1.45 grms. of the sub-nitrate and make the necessary solution up to 100 c.c.

**Method of Procedure.**—Pour 10 c.c. of the standard solution of bismuth into a 50 c.c. matrass, add 10 c.c. of the titrated solution of KI, and make up to 50 c.c. with—

Glycerin at 30° + distilled water, *q.s.*

In a second matrass of 50 c.c. capacity, place 0.15 gm. of the sub-nitrate of bismuth under examination dissolved in dilute nitric acid, add 10 c.c. of glycerin at 30°, then 10 c.c. of the titrated KI, and make up to 50 c.c. with—

Glycerin at 30° + distilled water, *q.s.*

There then only remains to make the colorimetric examination either by varying the thickness of the solution or by dilution. Bismuth in any form can always be brought to the state of bismuthic nitrate; therefore the above method is applicable to all the mineral or organic derivatives of bismuth.

It may be conceived that inversely it is possible to effect the titration of an iodide in solution, by means of a titrated solution of bismuth, so long as there is a large excess of glycerin present to prevent the precipitation of BiI<sub>3</sub>, as we remarked at the commencement of this paper.

Further, certain substances which have very definite relations with the iodides, may be estimated indirectly by this method.

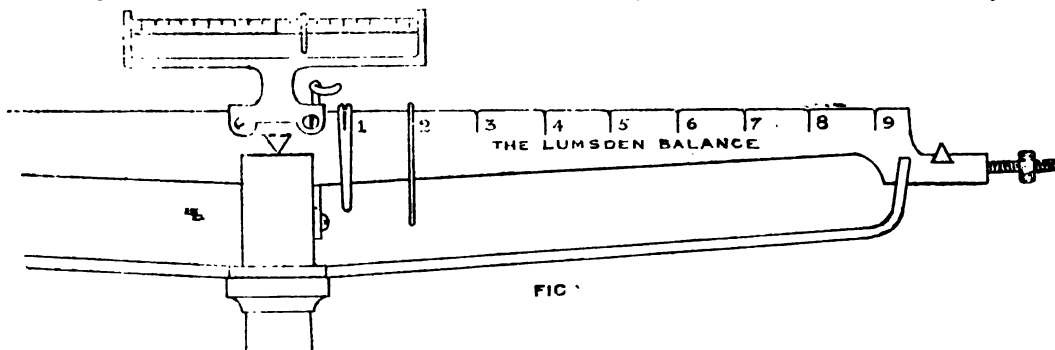
In a future paper we shall describe the method of working for the estimation of each individual bismuthic derivative, and inversely of the iodides and substances having precise relations with them.—*Journ. de Pharm. et de Chim.*, Series 6, vol. xviii., No. 9.



# NEW DESIGNS FOR CHEMICAL BALANCES.

By JOHN S. LUMSDEN D.Sc., Ph.D.

TEACHERS of elementary classes in chemistry and physics are greatly troubled by the destruction and loss of the smaller weights when students are performing experiments requiring the use of a balance. The balances used in schools and by beginners in colleges weigh accurately to a centigram., and with approximate accuracy to a milligram., yet many types have no graduations on the beams, and students must employ the very smallest weights. There is in consequence great expenditure of time and the duration of the usefulness of the milligram. weights is only a matter of days,



The annexed diagram (Fig. 1) shows a school balance, in the use of which no free weights smaller than a gram. are employed, the lesser values down to milligrams. being got by rider weights of a ring shape which are permanently kept on the balance.

The right arm of the beam has ten divisions, and decigrams. are found by a gram. rider, and centigrams. by a decigram. rider. These riders when not in use are supported on a hook which is screwed to the brass block on

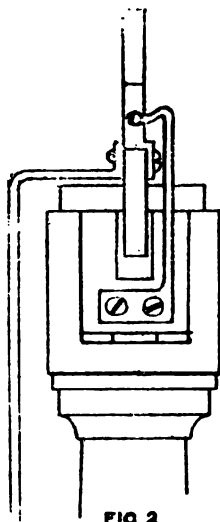


FIG 2

the top of the centre rod, as shown in Fig. 2. In order to make weighings such as 0.11 or 0.22 gram. possible, a notch is made on the upper part of the heavier rider, so that the lighter one, which has a greater diameter, may rest on the top of it. A light aluminium auxiliary beam, one-fifth of the length of the main beam, is used to measure milligrams. It is divided on each side of the centre into ten parts and

a 5 centigram. ring rider is employed, which rests on the central division when not in use. This auxiliary beam is an important feature. It ensures in every case that the student will attempt to weigh to a milligram., and a 5 centigram. rider is so strong that there is little danger of it being destroyed. If there is no objection to the use of decigram. weights, the two lighter riders, one on each beam, make a very simple system, and by having a loop on the riders, they could be moved when the balance is in a case by a sliding shifter.

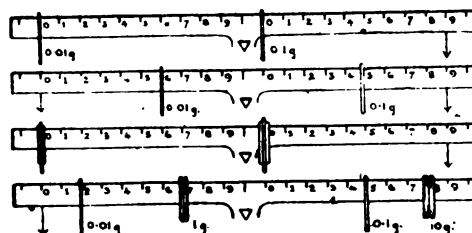
The use of a balance of this kind will effect a saving of time, it will be as accurate and only slightly dearer than the balances presently employed, and the weights will never get lost.

Mr. S. J. Shand, a student in the laboratory of Uni-

versity College, Dundee, has devised an ingenious method of graduating a balance which is of interest and may be of practical value.

In Fig. 3 a beam is shown graduated into ten parts on each side of the centre, but the numbers on both sides read from left to right. The first division on the right is marked 0 and the first division on the left is marked 9. A decigram. rider placed on 0 to the right balances a centigram. rider on 0 to the left. This is the position of the riders when the balance is at rest.

If the decigram. rider is moved along to division 5, that is,  $\frac{1}{2}$  from the centre, the other rider remaining as before, the weight is 0.05 gram. If now the centigram. rider is brought towards the right, the weight increases each division by 0.001 gram., and when the rider rests on 6, the weight is 0.056 gram., as shown on Fig. 4.



A more complete example is got by using four riders, viz.: 0.01, 0.1, 1, and 10 grms. The zero position is shown on Fig. 5 and a weight of 8.752 grms. on Fig. 6.

The substance to be weighed having been placed on the left pan of the balance, the 10 grms. rider on the right arm is moved along and found to be too heavy on 9; it is therefore placed on 8. The 1 gram. rider on the left arm is then shifted forward, and being too heavy on 8 it is placed on 7. Similarly the 0.1 gram. rider is placed on 5 to the right and the 0.01 gram. rider on 2 to the left. The reading taken in the order of the weights of the riders is thus 8.752 grms.

A balance graduated in this way and using centigram. and decigram. riders would be very good for assay work, and

weighing machines for ordinary commercial purposes might be devised to work with heavy riders instead of free weights.

University College, Dundee

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxvii., No. 22, November 30, 1903.

**Fusibility of Mixtures of Bismuth Protosulphide and Silver Sulphide, Bismuth Protosulphide and Antimony Sulphide.**—H. Pélabon.—When silver sulphide and bismuth protosulphide are melted together a homogeneous liquid is produced. The temperature at which solidification begins can be easily determined exactly. A curve of fusibility of such mixtures can be constructed, having for ordinates the temperature of solidification, and for abscissæ values corresponding to the proportion of the mass of silver sulphide to the total mass of the mixture. In the same manner the author examines mixtures of bismuth protosulphide and antimony sulphide with a view to determining the curve of fusibility.

**Influences Accelerating or Retarding the Action of Manganese considered as a Metallic Ferment.**—A. Trillat.—The author makes various experiments on the action of manganese employed as a metallic ferment. He finds that to render the metal active for the conditions under which he is working, the medium to be oxidised must contain an alkali or an alkaline earth salt. For the same quantity of alkali increasing proportions of manganese retard the action, as in the case of diastasic phenomena. The progress of the reaction can be hindered by the presence of traces of certain substances. Indeed, in order that manganese may produce the maximum effect in a medium in a given time a number of conditions have to be fulfilled.

**Systematic Alcoylation of Arsenic.**—V. Auger.—Up to the present time no method has been discovered of attaching one, two, or three alcoyl groups severally to the molecule of arsenic. Cabour's method consists in heating the metalloid with an alcoholic iodide. This immediately results in the formation of a mixture of tri- and tetra-substituted derivatives, whilst Meyer's method apparently only produces a single product, sodium methylarsinate. It is, however, in generalising this latter reaction that the author arrives at the systematic introduction of alcoholic groups into arsenic compounds.

**Separation of Iodine from Alkaline Halogen Salts of Iodine, Chlorine, and Bromine by the Transformation into Iodic Acid, and a Method of preparation of Pure Iodine.**—H. Baubigny and P. Rivals.—In order to separate iodine from a mixture of iodides, chlorides, and bromides, the authors take advantage of the easy oxidisability of iodine, and transform the whole of it into iodic acid, a stable and non-volatile body. They then successively separate the bromine and the chlorine by distillation. This method has given excellent results. Permanganate instantly oxidises iodides to iodates. The authors also evolve from this separation a method of obtaining perfectly pure iodine.

## MISCELLANEOUS.

**King's College, London.**—Evening Course in Bacteriology.—A course in Practical and Clinical Bacteriology will be held every Monday evening from 6.30 to 8.30, commencing Monday, January 11th, and ending Monday, March 8th. The course will consist of lectures, demonstrations, and practical work. In the latter, the members of the class will make for themselves permanent preparations of the chief pathogenic micro-organisms, and will carry out the principal manipulations employed in bacteriological investigations. The fee will be £3 3s., including all materials and the use of a

microscope. Names must be sent in as soon as possible to the Secretary, or to Prof. Hewlett, as the class will not be held if less than six students join. Special classes are formed for Institute of Chemistry examinations.

**ERRATUM.**—In last issue, p. 317, col. 2, line 6 from top, for "nickel and zinc" read "nickel and cadmium."

## MEETINGS FOR THE WEEK.

**MONDAY, 4th.**—Society of Chemical Industry, 8. "On the Defects of Uncarburetted Water-gas as Fuel for Laboratory Use," by Dr. Chikashigé. "The Rapid Estimation of Mercury by means of Hypophosphorous Acid," by B. F. Howard. "The Determination of Moisture in Nitro-glycerin Explosives," by Arthur Marshall.

**TUESDAY, 5th.** } Royal Institution, 3. (Christmas Lectures, adapted  
**WEDNESDAY, 7th.** } to a Juvenile Auditory). "On Extinct Animals,"  
**THURSDAY, 9th.** } by Prof. Ray Lankester. M.A., LL.D., F.R.S.

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| <b>METALLURGY</b> .. .. .  | C. O. BARNISTER, Assoc. R.S.M.                                           |
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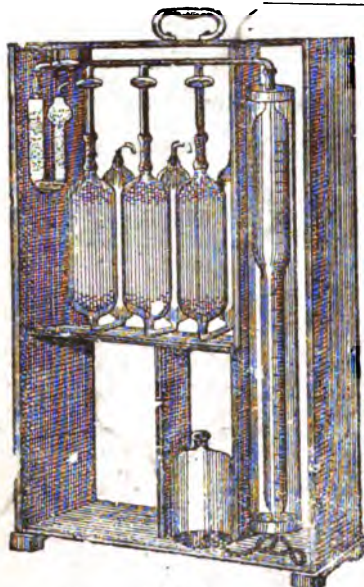
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### CONTENTS.

| ARTICLES:—                                                                                                                                 | PAGE |
|--------------------------------------------------------------------------------------------------------------------------------------------|------|
| On a Principal Cause of the Saltiness of the Dead Sea, by William Achroyd, F.I.C., &c.                                                     | 13   |
| Action of some Gases on Barium-ammonium, by M.M. Gunts and Mentzel                                                                         | 13   |
| The Determination of Benzene in Illuminating Gas, by L. M. Dennis and J. G. O'Neill                                                        | 14   |
| The Behaviour of Cerium, Lanthanum, Didymium, Neodymium, Praseodymium, Thorium, and Zirconium towards Organic Bases, by Burt Laws Hartwell | 15   |
| CHEMICAL SOCIETY                                                                                                                           | 17   |
| NOTICES OF BOOKS                                                                                                                           | 21   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                      | 22   |
| MISCELLANEOUS                                                                                                                              | 24   |

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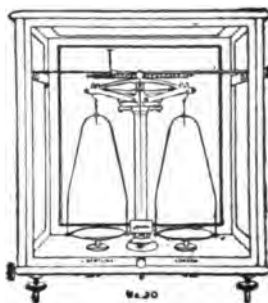
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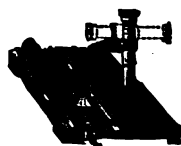
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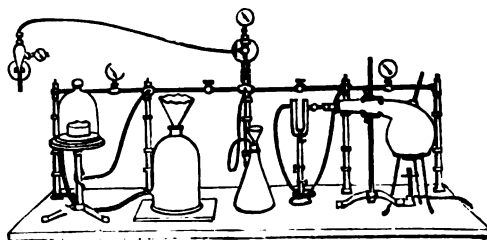
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2302.

## ON A PRINCIPAL CAUSE OF THE SALTNESSE OF THE DEAD SEA.\*

By WILLIAM ACKROYD, F.I.C., F.C.S., &c.,  
Public Analyst for Halifax.

THE saltness of the Dead Sea has been ascribed to two causes:—(1) The accumulation of chlorides derived from the rocks of the Holy Land by solvent denudation, and (2) the cutting off of an arm of the Red Sea by the rising of Palestine in past ages, with, in each case, the subsequent concentration of the solution by evaporation (Hull, *The Phys. Geol. of Arabia Petraea and Palestine*, pp. 119 and 120). There is a third cause, which is probably more important than either, viz., the atmospheric transportation of salt from the Mediterranean. The circulation of salt is a reality which must be taken into account. Brought from the sea by winds and falling in the rain, the salt is carried back to the sea by rivers, except in cases of inland lakes without outlet, where the saline solution remains for evaporation, and I have shown that in the case of an inland Pennine reservoir such a cause would produce a water as salt as that of the Dead Sea in a fraction of the time usually assigned to the Pleistocene Age (*Geol. Mag.*, October, 1901, p. 446; also compare J. G. Goodchild, *Trans. Geol. Soc. of Glasgow*, 1898, vol. xi., Part I., p. 84).

For the purpose of the present paper analyses of rain-water from the Holy Land are wanting; as, however, they are not at present available, I assume that the rain, like that of other lands, is charged with salt to a degree which varies in a direct manner with the velocity of the winds coming from the sea; it then only remains to show that the rocks are not abnormally salt bearing (*Proceedings of the Yorks. Geol. and Polytechnic Soc.*, vol. xiv., Part III., pp. 403–408).

I have had forwarded to me by the Palestine Exploration Fund specimens of the rocks on which Jerusalem is built as samples of Palestine rocks. They are limestones of various compositions, and the amount of common salt, calculated from the chlorine I have found in them, is given in the following table:—

| Description of limestone. | Per cent of chlorine. | Calculated per cent of common salt. |
|---------------------------|-----------------------|-------------------------------------|
| 1. Kakule .. .. .         | 0.025                 | 0.041                               |
| 2. Nahre .. .. .          | 0.001                 | 0.002                               |
| 3. Meleke .. .. .         | 0.006                 | 0.010                               |
| 4. Misse (yehudi) ..      | 0.005                 | 0.008                               |
| 5. Misse (helu) .. .      | 0.0015                | 0.002                               |
| 6. Misse (achmar) ..      | 0.001                 | 0.002                               |
| Average .. .. .           | —                     | 0.01                                |

The salt contained in these rocks, except in the case of Kakule limestone, is no greater in amount than that found in the limestones of other lands which similarly approximate to a general average of 0.01 per cent of chlorine. This amount of chlorine would be quite inadequate to account for the salt in the Dead Sea. By a technical argument, based on the amount of chlorine in a rock and its rate of denudation, I have shown that the salt yielded to rivers from this source is not a ninety-ninth of that which has been supplied by rain-water (CHEMICAL NEWS, vol. lxxxiii., p. 301, and *Geol. Mag.*, October, 1901, p. 447). Nor would the saltness of the Dead Sea be fully accounted for if a marine area had been cut off during the rising of the land, as the initial saltness thus acquired

would only be about a fourth of that subsequently attained to, and moreover in this condition of saturation it has been for an unknown length of time continuously precipitating its excess of salt. Hull observes (*op. cit.*, p. 120):—"The increase of saltness in the waters of the Dead Sea has probably been very slow, and dates back from its earliest condition when its waters stretched for a distance of about 200 miles from north to south. While the uprising of the land and the sinking down of the Jordan Arabah depression were in progress during the Myocene period, some of the waters of the outer ocean, themselves salt, were probably enclosed and retained; but from the occurrence of the shells in the marls in the Arabah Valley, it would appear that when the waters of the great inland lake were at their maximum elevation, they were sufficiently fresh to allow of the presence of molluscos life. This would be during the Pluvial epoch, but at the stage represented by the salt beds of Jebel Usdum, the waters, which were then 600 feet higher than at present, must have been saturated with chloride of sodium." One may add that the intensity of meteorological conditions in the past geological history of Palestine have been much more severe than those now obtaining, and the atmospheric transportation of salt would be correspondingly greater (Tristram, "The Land of Israel," p. 320). Some of the salt then accumulated has been left by the dwindling waters of the Dead Sea in areas to the north and south, notably in Jebel Usdum, and the highly brackish rivulets which come from these neighbourhoods now are but contributing again what long ago came from more distant sources.

I find confirmation of the theory in the fact that the ratio of the chlorine to the bromide in the waters of the Dead Sea is approximately the same as that for these two elements in the Mediterranean Sea (*Proceedings of the Yorks. Geol. and Polytechnic Soc.*, 1902, vol. xiv., Part III., p. 408).

In conclusion, I have to offer my best thanks to Mr. Walter Morrison, J.P., of Malham, and Mr. George Armstrong for the privilege of having been enabled to examine the rocks from Palestine which are mentioned in this paper.

## ACTION OF SOME GASES ON BARIUM-AMMONIUM.

By MM. GUNTZ and MENTREL.

WE have examined the action of several gases on the solution of barium-ammonium in liquid ammonia, notably that of oxygen, of carbonic oxide, and of binoxide of nitrogen.

The pure dry gas with which the experiment is to be made, is collected in a mercury gas-holder, and for the purpose of complete desiccation it remains in contact with phosphoric anhydride contained in a tube, T, connected with a manometer. By pouring mercury into a branch tube of the gas-holder, any desired pressure can be attained, and the stop-cock turned off to retain it.

To carry out an experiment, the solution of barium-ammonium is prepared in a suitable vessel, A, cooled to -50°, to bring the tension of the ammonia below that of the gas contained in the tube, T, and the tap is opened, making communication between the tube T and the vessel A. When a sufficient quantity of gas is estimated to have passed, the tap is turned off. The volume of T having been previously determined, it suffices to read off the pressure on the manometer before and after the introduction of the gas into the vessel, to calculate the actual quantity brought into the presence of the ammonium.

When the reaction is terminated, the vessel, A, is allowed to return to the ordinary temperature; the liquid ammonia volatilises and escapes by a side tube, carrying off also the gases which have not reacted, or those which may have been produced during the experiment. It is easy to collect these gases for analysis.

\* From the Quarterly Statement of the Palestine Exploration Fund.

*Action of Oxygen.*

By bringing oxygen into contact with the solution of barium-ammonium prepared in a small flask, this gas is absorbed; it produces a white gelatinous precipitate, remaining in suspension in the liquid ammonia. The reaction is slow at  $-50^{\circ}$ , and the last portions of the barium-ammonium are very difficult to decompose on account of its slight solubility in ammonia at this temperature. The gelatinous precipitate formed encloses the non-dissolved ammonia in bubbles, and prevents it coming in contact with the oxygen.

At a higher temperature, and especially when operating with an excess of oxygen, the blue solution is decolorised very rapidly.

When the reaction is complete, the ammonia is entirely removed; a white amorphous powder is then deposited, which, when treated with dilute hydrochloric acid, is dissolved without the evolution of any gas, giving a solution containing peroxide of hydrogen and no ammonia.

Whatever the method of procedure adopted, and the temperature of the experiment, the products obtained have practically identical compositions.

The barium was weighed before the reaction; the peroxide formed is estimated by permanganate, and the active oxygen in the solution calculated into binoxide of barium,  $\text{BaO}_2$ .

At  $-50^{\circ}$ , by adding oxygen gradually as it is absorbed, and stopping the experiment after the decolorisation of the solution, we find 9.07 of barium in the form of  $\text{BaO}_2$  and 90.9 in the form of  $\text{BaO}$ .

At  $-35^{\circ}$ , the oxygen being in excess and remaining in contact with the gelatinous product after decolorisation, the composition was 7.5 of barium in the form of  $\text{BaO}_2$  and 92.5 in the form of  $\text{BaO}$ .

Thus, contrary to what takes place with the alkaline ammonias, the reaction of the oxygen on the barium only gives a small quantity of the higher oxide.

*Action of Carbonic Oxide.*

The action of carbonic oxide on barium varies according to the conditions of the experiment.

When we heat the metal in a current of this gas, no attack takes place below  $500^{\circ}$ . At this temperature it becomes black, but the change is only superficial. The black layer which covers the metal gives off acetylene in the presence of water; thus there is a formation of carbide of barium.

The action of carbonic oxide on the ammoniacal solution of barium-ammonium is quite different. At  $-50^{\circ}$  it takes place very easily, and the decolorisation is even more rapid than with oxygen. A yellow gelatinous precipitate is formed, which is deposited, on the removal of the ammonia, in the form of a yellow amorphous powder.

We determined the composition of this body by weighing the substance formed by the fixation of the carbonic oxide by a known weight of barium; this is easily done. Our results were:—

|          | Found | Theory for $\text{Ba}(\text{CO})_2$ . |
|----------|-------|---------------------------------------|
| Ba .. .. | 72.1  | 71.05                                 |
| CO .. .. | 27.9  | 28.95                                 |

We have not been able to make an analysis of the sub-carbonate of barium, as this body decomposes with loss of heat, if only a little air or a trace of moisture is allowed to enter the vessel containing it.

The product of this decomposition is completely soluble in water, and gives a yellow solution that we have not yet examined.

If barium sub-carbonate is heated *in vacuo*, it becomes brown at about  $100^{\circ}$ ; at about  $250^{\circ}$  it decomposes with incandescence, the mass swells up, but without any evolution of gas or explosion. The residue consists of baryta, carbonate of barium, and finely divided carbon.

The product which has undergone the action of the air is also decomposed, but at a higher temperature, giving the same substances.

The reaction of the decomposition of the sub-carbonate of barium may, consequently, be expressed in the same manner as that of the sub-carbonate of sodium,  $2\text{Ba}(\text{CO})_2 = \text{BaO} + \text{BaCO}_3 + 3\text{C}$ .

*Action of Binoxide of Nitrogen.*

Binoxide of nitrogen reacts easily on barium-ammonium. The gelatinous precipitate produced by this reaction is deposited in the form of a white powder on the evaporation of the ammonia.

We have observed the formation of the hyponitrite of barium,  $\text{Ba}(\text{NO})_2$ ; this was recognised by its silver salt, after having allowed the white powder to become slowly hydrated by contact with steam before dissolving it, as it decomposes with the evolution of protoxide of nitrogen in the presence of a drop of water.

Thus the solution of barium-ammonium behaves like that of sodium.

We intend proceeding with the examination of the other compounds which may be obtained by means of this solution.

We may add that barium-ammonium can be also prepared by the action of liquid ammonia on 60 per cent amalgam. In this case there remains an amalgam very poor in barium.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 12.

## THE DETERMINATION OF BENZENE IN ILLUMINATING GAS.\*

By L. M. DENNIS and J. G. O'NEILL.

IN 1891, Hempel and Dennis described a method for the volumetric determination of certain hydrocarbons that are usually present in illuminating gas (*Ber. d. Chem. Ges.* xxiv., 1162). Up to that time, all hydrocarbons in this product, with the exception of methane, had been determined by absorption with fuming sulphuric acid, and had been classed under the general term "heavy hydrocarbons." It is true that Bunsen ("Gasometrische Methoden," Second Edition, 1877, p. 142) gives an analysis of illuminating gas in which the percentages of benzene, ethylene, and propylene are stated, but the amounts of these three gases were calculated by means of equations from the results of explosions with air and oxygen, and the calculation was based upon the assumption that the heavy hydrocarbons in the gas consisted only of ethylene, propylene, and benzene. It was ascertained by Hempel and Dennis that certain hydrocarbons, such as benzene and naphthalene, could be removed, in part at least, by means of absolute alcohol, the remainder of the heavy hydrocarbons being then absorbed by fuming sulphuric acid, and the methane being finally determined by explosion or combustion. In 1894, Noyes and Blinks adapted the method of Hempel and Dennis to the Bunte burette (*Journ. Am. Chem. Soc.*, xvi., 697).

Recent work in this laboratory, however, has shown that while the absorption of benzene by means of alcohol may sometimes give agreeing results, the removal of the benzene is usually by no means complete, and the results, at times, show wide variations. The following analyses are given in confirmation of this statement. Air was drawn into a Hempel burette containing water as the confining liquid, and was measured. It was then passed into a gas pipette containing benzene, and was drawn back into the burette, and the increase in volume noted. The mixture of benzene and air was next passed into a pipette containing mercury and 3 c.c. of absolute alcohol, and was shaken for three minutes in contact with the alcohol. The residue was then drawn back into the burette, and was passed into a pipette filled with water, and was shaken with that liquid for three minutes to remove the vapour of

\* From the *Journal of the American Chemical Society*, xxv., No. 5.



alcohol. The gas was now drawn back into the burette and measured. The results were :—

TABLE I.

|                                                       | I.   | II.  | III. |
|-------------------------------------------------------|------|------|------|
|                                                       | C.c. | C.c. | C.c. |
| Air taken . . . . .                                   | 67.0 | 50.0 | 52.2 |
| Air plus benzene (C <sub>6</sub> H <sub>6</sub> ) . . | 73.0 | 54.0 | 57.0 |
| After shaking with alcohol . .                        | 69.0 | 51.8 | 54.2 |
| After shaking with water . .                          | 68.4 | 51.4 | 53.7 |
| Benzene taken . . . . .                               | 6.0  | 4.0  | 4.8  |
| Benzene found . . . . .                               | 4.6  | 2.6  | 3.3  |

Experiments were next tried to ascertain whether repeated treatment with alcohol would remove all of the benzene, and, as will be seen from the table below, it appears that this reagent is unable to remove benzene completely from mixtures of that substance with air.

TABLE II.

|                                                                                                        | I.   | II.  | III. | IV.  | V.   |
|--------------------------------------------------------------------------------------------------------|------|------|------|------|------|
|                                                                                                        | C.c. | C.c. | C.c. | C.c. | C.c. |
| Air taken . . . . .                                                                                    | 58.6 | 60.1 | 63.0 | 56.8 | 67.9 |
| Air plus benzene . . . . .                                                                             | 62.8 | 64.6 | 67.0 | 61.0 | 72.8 |
| After shaking with alcohol . .                                                                         | 59.8 | 61.6 | 63.8 | 57.8 | 69.0 |
| After shaking with water . .                                                                           | 59.4 | 61.1 | 63.8 | 57.6 | 68.8 |
| After second shaking with alcohol . . . . .                                                            | 59.2 |      |      | —    | —    |
| After second shaking with water . . . . .                                                              | 59.1 |      |      | —    | —    |
| After third shaking with alcohol . . . . .                                                             | 59.1 |      |      | —    | —    |
| After third shaking with water . . . . .                                                               | 59.1 |      |      | —    | —    |
| After passing residue into fuming sulphuric acid and then into the potassium hydroxide pipette . . . . | 58.7 | 60.2 | 62.9 | —    | —    |

It was therefore apparent from these results that for the complete and speedy removal of benzene from illuminating gas some absorbent other than absolute alcohol must be employed.

In 1897, Hofmann and Küspert described (*Zeit. Anorg. Chem.*, xv., 204) certain compounds of hydrocarbons with metallic salts, and stated that when illuminating gas acts upon a mixture of nickel hydroxide and aqua ammonia, there results a compound of nickel cyanide with ammonia and benzene, Ni(CN)<sub>2</sub>.NH<sub>3</sub>.C<sub>6</sub>H<sub>6</sub>. This statement led the authors of the present paper to examine into the action of an ammoniacal nickel solution upon benzene with the view to ascertaining whether a method could be developed for the volumetric determination of the benzene that is present in the form of vapour in gas mixtures. Sufficient of the absorbent to fill a Hempel simple absorption pipette (about 150 c.c.) was prepared by dissolving 25 grms. of crystalline nickel nitrate, Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O, in 50 c.c. of water and adding 50 c.c. of strong aqua ammonia. The solution was allowed to cool, was decanted from any salt that separated out, and strong aqua ammonia was then added until the volume amounted to 150 c.c. The solution loses its efficiency if diluted with water, and its absorptive power is greatly diminished if nickel hydroxide is present in suspension. The analytical results given in this paper were obtained with the reagent prepared in this manner. Experiments showed that this solution absorbed nothing from air, but that when shaken with a volume of air it gave off a small amount of ammonia gas. In the first series of experiments it was sought to remove this ammonia by passing the gas mixture into a Hempel pipette filled with water, but, since this did not entirely remove the ammonia, it was found necessary to use in place of the water a 5 per cent solution of sulphuric acid.

Later experiments made in this laboratory by Mr. W. C.

Geer have shown that it is possible to prepare the reagent in such manner as to do away with the necessity of the treatment of the gas mixture with dilute sulphuric acid. The method of preparation of the reagent is as follows :—Forty grms. of nickel nitrate are dissolved in 160 c.c. of water to which has been added 2 c.c. of nitric acid of specific gravity 1.44. This solution is poured slowly with constant stirring into 100 c.c. of ammonium hydroxide, specific gravity 0.908. The resulting deep blue solution is used in the absorption. It has a very slight tinge of lavender. The odour of ammonia is noticeable, but is not strong. The few analyses that have been made with the reagent prepared in this manner indicate that it is similar in action to that prepared as above described, and that it is equally efficient. It should, however, be more thoroughly tested before complete reliance is placed upon it.

(To be continued),

# THE BEHAVIOUR OF CERIUM, LANTHANUM, NEODYMIUM, PRASEODYMIUM, THORIUM, AND ZIRCONIUM TOWARD ORGANIC BASES.\*

By BURT LAWS HARTWELL.

AN investigation along these lines seemed to promise well because of the success which Jefferson met while prosecuting a similar study with a limited number of aromatic bases (*Journ. Am. Chem. Soc.*, xxiv., 540). It was hoped that some organic base would be found, among the large number to be tried, which would lead to the separation of thorium and zirconium, or failing in this, that a method would be disclosed by which these two elements could be separated from cerium, lanthanum, neodymium, and praseodymium.

Large quantities of thorium and zirconium nitrates were prepared from the mineral thorite and zircon. Numerous qualitative tests were made and the solvents for the bases were, in most cases, water and alcohol, or mixtures of these two, depending upon the solubility of the base. In some instances ether was added to advantage. Alcohol, above a certain strength, precipitated the thorium and zirconium nitrate solutions, so that this point had to be constantly considered during the testing. The salt solution was usually added in small quantities at a time to the solution containing a liberal amount of the organic base. Heat was applied, if precipitation did not occur in the cold. The solutions were not permitted to stand for long periods, as it was thought that differences which did not manifest themselves readily, under ordinary conditions, would scarcely indicate the probability of practical quantitative separations.

The bases studied in this investigation were obtained from Kahlbaum. Those employed by Jefferson were usually omitted here (*loc. cit.*).

The bases of the first and second groups were manifestly unsuited for the purpose in mind. Those of the third and fourth groups showing differences in deportment with the several elements under consideration were given more particular attention, while those which were insoluble in dilute alcoholic solutions were placed to one side. The following paragraphs record the observations made with the several bodies which seemed best adapted for quantitative separations.

**Thorium and Zirconium with the Chloranilines.**—The different behaviours of the chloranilines substantiate the views held regarding their relative basicity (van 't Hoff's "Lectures on Theoretical and Physical Chemistry," vol. ii., p. 102). *o*-Chloraniline did not precipitate thorium or zirconium. *m*-Chloraniline precipitated zirconium in the cold, but with thorium heat was required to produce

\* Contributions from the John Harrison Laboratory of Chemistry. (From author's thesis for Ph.D. degree). From the *Journal of the American Chemical Society*, xxv., No. 11.

*Reagents which Produced no Precipitate with Salts of any one of the Six Elements.*

|                                 |                          |
|---------------------------------|--------------------------|
| Benzylaniline.                  | <i>o</i> -Nitrilaniline. |
| Dimethylnitrosamine.            | <i>o</i> -Chloraniline.  |
| <i>p</i> -Nitrilaniline.        | Piperine.                |
| <i>p</i> -Nitrophenylhydrazine. | Succinimide.             |
| Dipropylnitrosamine.            | Tetranitromethylaniline. |
| <i>m</i> -Nitrilaniline.        |                          |

*Reagents which Caused Precipitates with Salts of all Six of the Elements.*

|                    |                                |
|--------------------|--------------------------------|
| Allylamine.        | Monoamylamine.                 |
| Benzylmethylamine. | Monethylamine.                 |
| Bornylamine.       | Monomethylamine.               |
| Camphylamine.      | Monopropylamine.               |
| Diamylamine.       | Neurine.                       |
| Dibenzylamine.     | Normal butylamine.             |
| Diethylamine.      | Normaldibutylamine.            |
| Dimethylamine.     | Propylenediamine.              |
| Dipropylamine.     | Tetraphylammonium hydroxide.   |
| Ethylenediamine.   | Tetramethylammonium hydroxide. |
| Heptylamine.       | Triethylamine.                 |
| Hexylamine.        | Trimethylamine.                |
| Isobutylamine.     | Tripropylamine.                |
| Isotributylamine.  |                                |

*Reagents which Precipitated only Thorium and Zirconium.*

|                                 |                             |
|---------------------------------|-----------------------------|
| Benzidine.                      | Isoquinoline.               |
| <i>m</i> -Bromaniline.          | $\alpha$ -Picoline.         |
| <i>p</i> -Bromaniline.          | <i>p</i> -Toluidine.        |
| <i>p</i> -Bromophenylhydrazine. | <i>m</i> -Toluylenediamine. |
| <i>p</i> -Chloraniline.         | Tribenzylamine.             |

*Reagents not Included in the Preceding Groups and whose Reactions are mentioned upon Succeeding Pages.*

|                          |                         |
|--------------------------|-------------------------|
| <i>m</i> -Chloraniline.  | $\beta$ -Naphthylamine. |
| Diethylaniline.          | Tetrahydroquinoline.    |
| Hexamethylenetetramine.  | <i>m</i> -Toluidine.    |
| Monethylaniline.         | <i>o</i> -Xylidine.     |
| Monomethylaniline.       | <i>p</i> -Xylidine.     |
| $\alpha$ -Naphthylamine. |                         |

precipitation, while *p*-chloraniline precipitated the solutions of both elements, even in the cold. Solutions of cerium, lanthanum, neodymium, and praseodymium salts were not affected by these reagents.

*Zirconium and Thorium with m-Chloraniline.*—Much work was done upon *m*-chloraniline to arrive at the proper conditions in the strength of alcohol and temperature favourable to the complete precipitation of the zirconium solutions, and at the same time to leave the thorium salt unprecipitated. A solution containing equal parts of water and commercial alcohol was found most satisfactory. At a temperature of 60–70°, this solvent containing *m*-chloraniline occasioned no precipitation in solutions of thorium nitrate, while in zirconium nitrate solutions it precipitated 0.0995 grm., 0.0988 grm., 0.0990 grm., and 0.0988 grm. of zirconium oxide, instead of 0.0995 grm. obtained by ammonia or 0.0993 grm. by direct evaporation and ignition. On repeating the experiment with a mixture of the thorium and zirconium nitrates, both elements were completely precipitated.

*Thorium and Zirconium with Hexamethylenetetramine (Formin).*—Formin in the qualitative tests showed a marked difference in behaviour. Thus a thorium solution, after its addition, stood forty-two hours at room temperature without the appearance of a precipitate. Upon adding the zirconium nitrate solution, both hydroxides were precipitated completely,

$\alpha$ - and  $\beta$ -Naphthylamine, *m*-bromaniline, and *p*-bromophenylhydrazine precipitated zirconium solutions much more completely than those of thorium, but separations could not be effected by means of them. They reacted

indifferently with the salts of cerium, lanthanum, neodymium and praseodymium. *o*-Bromaniline was not tried at all in this investigation.

In a solution of the nitrates of zirconium and cerium, *m*-chloraniline precipitated 0.0532 grm. of zirconium oxide, instead of 0.0539 grm. obtained with ammonia water. *p*-Chloraniline precipitated 0.0536 grm., *m*-bromaniline 0.0524 grm., and  $\beta$ -naphthylamine 0.0533 grm. In the first three filtrates 0.0396 grm., 0.0400 grm., and 0.0396 grm. respectively, of cerium oxide were obtained, instead of the theoretical 0.0389 grm. In using *p*-chloraniline with a solution containing twice the quantities of zirconium and cerium oxide given above, a very satisfactory quantitative separation resulted.

In precipitating a mixture of zirconium and cerium salts with *p*-toluidine, 0.1341 grm. of zirconium oxide was found, while the theoretical amount present was 0.1347 grm. This same reagent added to a solution of thorium and cerium nitrates precipitated 0.1246 grm., 0.1236 grm., and 0.1261 grm., instead of 0.1247 grm.

While monomethylaniline precipitated cerium solutions completely and apparently had no marked effect upon those of lanthanum, neodymium, and praseodymium, the attempts at quantitative separations were fruitless. Diethylaniline precipitated cerium salts quite completely. The insolubility, however, of this reagent compelled the use of such large amounts of alcohol that serious difficulties arose. These were not overcome. A quantitative separation of cerium from lanthanum was made by means of tetrahydroquinoline. Two precipitations of the cerium were necessary.

*o*- and *p*-Xylidine, as well as monethylaniline, precipitated cerium completely from its salt solutions. *m*-Toluidine seemed inadequate for this purpose.

(To be continued).

*Transactions of the American Electrochemical Society.*—Fourth General Meeting, Niagara Falls, N.Y., September 17, 18, and 19, 1903. (Vol. iv.).—At the above meeting of the Society a number of interesting papers were read, among them being "Advances in Electro-metallurgy of Iron Production," by Marcus Ruthénburg; "Note on the Electro-metallurgy of Gold," by W. H. Walker; "Electrolytic Copper Refining," by W. D. Bancroft; "Efficiency of the Nickel-plating Tank," by O. W. Brown; "A New Type of Electrolytic Cell," by P. G. Salom; "On the supposed Electrolysis of Water Vapour," by F. A. Lidbury; "Theoretical Properties of Free Ions in Solutions," by E. F. Roeber; "Present Status of the Electrolytic Dissociation Theory," by W. D. Bancroft; and a general discussion of the "Theory of Electrolytic Dissociation."

*Simplification of the Method for the Estimation of Zinc in the Form of Sulphide.*—A. Thiel.—The method recommended by the author has for its object the elimination of the always long and tedious filtration of the sulphide. The solution of zinc is precipitated in the usual manner by sulphuretted hydrogen in the presence of acetate of ammonia. Boil for two minutes by direct heating over a gas flame. After the deposition of the precipitate, the clear liquid is decanted on to an ordinary filter. The residue is washed several times with boiling water charged with sulphuretted hydrogen; it is then passed into an Erlenmeyer flask of 50 c.c. capacity, and evaporated on the water-bath. The neck of the flask should be kept plunged in the steam. The ash of the filter is added to the residue, and the whole heated to 120° in a current of sulphuretted hydrogen; the operation is terminated by driving off the last traces of sulphur in a current of hydrogen. If the solution available for the precipitation of the zinc contains a large amount of alkali or alkaline earths, the precipitate should be re-dissolved, and the operation of precipitation repeated a second or even a third time. The results are very exact.—*Zeit. Anorg. Chem.*, vol. xxxiii., p. 1.

# PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, December 16th, 1903.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MESSRS. E. F. ARMSTRONG, H. J. W. BLISS, E. R. HUGHES, B. INGRAM, and C. MÜLLER were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Alick Cole Benson, 33, Perham Road, West Kensington; Robert Gawler, M.Sc., 3, South View Terrace, Leeds; Leo Frank Guttman, Ph.D., 18, Aberdare Gardens, N.W.; Cyril Douglas McCourt, 52, Victoria Road, Clapham, S.W.; Benjamin L. Murray, 19, University Place, New York; Thomas Stewart Patterson, Ph.D., Lyddon Hall, Leeds; Alfred Shrubsole, 91, Holyhead Road, Coventry; Samuel John Smith, 41, Grafton Street, Dublin; Alfred Tingle, Ph.D., B.Sc., Shantung, China.

Of the following papers, those marked \* were read.

\*165. "The Relative Strengths of the Alkaline Hydroxides and of Ammonia as Measured by their Action on Cotarnine." By J. J. DOBBIE, A. LAUDER, and C. K. TINKLER.

The authors have shown (*Trans.*, 1903, lxxxiii., 598) that in aqueous solution, cotarnine has the "ammonium hydroxide" form, whereas in ethereal or chloroform solution it exists as a "carbinol."

These two forms are distinguished by having widely different and very characteristic absorption spectra (*loc. cit.*, Plate I., carbinol form; Plate II., ammonium hydroxide form).

When cotarnine in aqueous solution is acted on by sodium hydroxide or any other soluble base, it is converted into the carbinol form. The proportion of the cotarnine which undergoes this change depends on the amount of sodium hydroxide present, the change being practically complete when a milligram-molecule of cotarnine is dissolved in one litre of normal sodium hydroxide solution. After the addition of any given quantity of sodium hydroxide, a state of equilibrium is established almost instantaneously, and no further change takes place in moderately dilute solutions, even after several hours, provided that the temperature is kept constant. Each additional quantity of sodium hydroxide causes a further transformation of "ammonium hydroxide" into "carbinol" form. This change can be followed through all its phases by photographing the spectra of the solution after each addition of sodium hydroxide, the spectra obtained being in reality those of mixtures of the two modifications (*loc. cit.*, Plate III., Figs. 17-24), the relative amounts of which can be determined by comparing these photographs with a standard series obtained by photographing the spectra of solutions prepared by mixing the two forms of cotarnine in definite proportions. It is possible, therefore, to estimate the amount of change which any given quantity of sodium hydroxide produces in an aqueous solution of cotarnine, and to obtain a curve representing the effect of successive additions of the alkali.

The action of ammonia and of the hydroxides of lithium, sodium, potassium, barium, and calcium has been examined in this way. The curves for the first three hydroxides are nearly identical, from which it is inferred that, so far as their action on cotarnine is concerned, they have practically the same strength; those of the last two agree with one another, but differ slightly from the others, indicating a slight inferiority of strength, especially for the more concentrated solutions.

The curve for ammonia, which differs widely from those of the alkaline hydroxides, indicates that this base is much weaker than the latter substances.

### DISCUSSION.

Mr. W. C. REYNOLDS expressed the hope that Prof. Dobbie would be able to investigate, in this connection, the

parent bases, hydrastine and narcotine, from which hydrastine and cortamine are derived by oxidation. In the case of hydrastine, he had noticed the existence of two modifications, one variety melting at about 131-132°, and the other at a temperature somewhat above 140°. If either variety was dissolved in alcohol or acetone and the solvent removed at the temperature of the water-bath, the less fusible variety was obtained; but if the solution was allowed to evaporate spontaneously, or if a concentrated hot solution was cooled and then allowed to crystallise, the more fusible variety was obtained. The less fusible variety was always produced by the fractional precipitation by ammonia of the hydrastine salts.

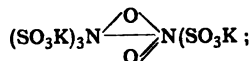
Moreover, if the temperature when in the neighbourhood of the melting-point was raised very slowly (about 1° per half-hour), the crystals appeared to change from the more fusible to the less fusible modification. The crystals which melted at 131-132° when heated in the ordinary manner, did not soften until the temperature was considerably above 140°, and then melted somewhat indefinitely.

Under similar conditions, both varieties seemed to have the same specific rotation in solution.

Similar changes were not observed in the case of the allied alkaloid, narcotine.

\*166. "Peroxyaminesulphonates and Hydroxylaminetrisulphonates (Sulphazilates and Metasulphazilates)." By T. HAGA.

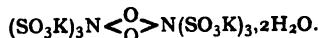
By oxidising potassium hydroxylaminedisulphonate, Fremy obtained the salts which he called sulphazilate and metasulphazilate. Claus re-named the former "oxysulphazotate," and formulated its constitution as—



the latter he called "trioxyazotate," giving it the formula  $\text{O}:\text{N}(\text{SO}_3\text{K})_3, \text{H}_2\text{O}$ . Raschig changed these formulæ into—

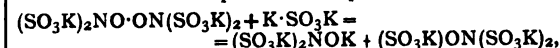


and—



whilst Hantzsch and Semple have re-named the sulphazilate "nitroxydisulphonate" with the formula,  $\text{O}:\text{N}(\text{SO}_3\text{K})_2$ , and have gone back to Claus's formula for the metasulphazilate.

The author has found that (1) potassium sulphazilate interacts with normal potassium sulphite—



to form normal potassium hydroxylaminedisulphonate and potassium metasulphazilate; (2) the sulphazilate decomposes spontaneously into the metasulphazilate, hydroxylaminedisulphonate, and much nitrous acid, the last being preserved as nitrite when potassium hydroxide is present,—

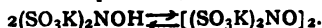


(3) potassium metasulphazilate is decomposed quantitatively into sulphate and normal aminedisulphonate (imino-sulphate) by sodium amalgam or by the zinc-copper couple,  $\text{KO} \cdot \text{SO}_2 \cdot \text{O} \cdot \text{N}(\text{SO}_3\text{K})_2 + 2\text{Na} = \text{KO} \cdot \text{SO}_2 \cdot \text{ONa} + \text{NaN}(\text{SO}_3\text{K})_2$ ;

and (4) the ultimate products of its hydrolysis, when heated with hydrochloric acid, are hydroxylamine and sulphuric acid.

The molecular magnitude of sodium hydroxylaminetrisulphonate, and also of the 2/3-normal sodium hydroxylaminedisulphonate, as determined by Loewenherz's method, is in each case that which contains only one atom of nitrogen. The constitution of the sulphazilates is therefore that expressed by the name, potassium peroxyaminesulphonate, and the formula

$\text{SO}_3\text{K})_2\text{NO}\cdot\text{ON}(\text{SO}_3\text{K})_2$ , and of the metasulphazilates that expressed by the name and formula, potassium hydroxylaminetrisulphonate,  $(\text{SO}_3\text{K})_2\text{N}\cdot\text{O}(\text{SO}_3\text{K})$ . The sulphazilates are oxime peroxides or peroximes, being produced by oxidising hydroxylaminedisulphonates with a variety of reagents (including ozone), even in the cold; they behave as oxidising agents, becoming again reduced to hydroxylaminedisulphonates,—



A metasulphazilate is the only known case of a triacylated hydroxylamine. Having, as a hydroxylaminetrisulphonate, one of its sulphonate radicles in union with oxygen, it is clearly one-third sulphatic, yet without being actually a sulphate. It is a mixed oxide or anhydride of two acid salts, one being the acid sulphate, and the other the 2/3 normal hydroxylaminedisulphonate,—



The inorganic mixed anhydride most closely analogous to a hydroxylaminetrisulphonate is Pelouze's salt, potassium hyponitrosulphate,  $(\text{SO}_3\text{K})\cdot\text{O}(\text{N}_2\text{OK})$ , the two salts agreeing in being stable in presence of caustic alkali, and in not yielding barium sulphate with a soluble barium salt, whilst giving rise to a sulphate with sodium amalgam or zinc. To understand this behaviour, it is only necessary to assume that the metasulphazilate ionises into three metallic kations, and a complex trivalent anion which includes within itself the sulphate radicle. Dunstan and Goulding have shown that zinc and acid reduce trialkylhydroxylamines to trialkylamines, so that if, as has hitherto been supposed, metasulphazilates were oxamines, they should reduce to aminetrisulphonates (nitrosulphates) and not to aminedisulphonates and sulphates. The author concludes that the nitrogen of the sulphazilates and metasulphazilates is exclusively tri- and not quinquevalent, as suggested by the earlier investigators of these salts.

There is so much difference between the properties of a peroxyaminesulphonate and those of the bluish-violet substance produced by the action of sulphur dioxide on a solution of nitrososulphuric acid in sulphuric acid that it is improbable that Sabatier's suggestion will prove correct as to the latter compound being the acid of Fremy's bluish-violet salt.

Potassium hydroxylaminetrisulphonate is shown to have 3/2 mols. of water of crystallisation, whereas Claus, who stated that the salt contained 1 mol. of water, was perhaps unaware that some of its water of crystallisation becomes fixed by hydrolysis during the drying. Sodium, ammonium, and hydroxy-lead hydroxylaminetrisulphonates have been prepared for the first time.

\*167. "Peroxylaminesulphonic Acid." By E. DIVERS.

In support of Sabatier's assumption that the bluish-violet colour caused by the action of sulphur dioxide on sulphuric acid containing nitrososulphuric acid is due to the formation of the unknown acid of Fremy's bluish-violet potassium salt (sulphazilate or peroxyaminesulphonate), it is pointed out that the difference in properties observed by Haga (preceding note) is hardly greater than that between the behaviour of nitrous acid in the respective forms of nitrososulphuric acid and potassium nitrite.

Sabatier's observations are shown to be quite consistent with the view that the acid is peroxyaminesulphonic acid; that is, a peroxide and a compound of trivalent nitrogen.

\*168. "Constitution of Nitric Peroxide." By E. DIVERS.

Haga's examination of Fremy's sulphazilate has established that it is *peroxylaminesulphonate*, a trivalent nitrogen compound and a peroxide. It is a sulphonated nitric peroxide, as suggested by Hantzsch and Semple, and when decomposed by water, gives a complex anhydrosulphate (hydroxylaminetrisulphonate), on the one hand, and, on the other, nitrous acid and sulphonated nitrous acid (hydroxylaminedisulphonate), equivalent respectively to the nitric and nitrous acids which are yielded by nitric

peroxide. Dinitric peroxide is therefore a true peroxide, *nitrosyl peroxide*,  $(\text{NO})_2\text{O}_2$ .

Mononitric peroxide must therefore be regarded as  $\text{O}:\text{N}:\text{O}$ , formulated with a univalent oxygen atom, and not with its nitrogen atom in the quadrivalent condition,  $\text{O}:\text{N}:\text{O}$ , as suggested by Piloty and Schwerin (*Ber.*, 1901, xxxiv., 1884 and 2354). For it seems appropriate to consider the constitution of the two forms of nitric peroxide to be the same, the only difference being the presence in the one form of the bivalent double atom of oxygen, and in the other form of a single univalent oxygen atom. A true peroxide is correctly defined as a compound in which some or all of the oxygen is exerting on the rest of the compound only half its usual valency. Piloty and Schwerin's *porphyreride*,  $(\text{C}_5\text{H}_9\text{N}_3):\text{NO}$  (*loc. cit.*) is to be regarded as being such a peroxide.

169. "Halogen Derivatives of Diphenyl and Dihydroxydiphenyl." By J. C. CAIN.

The author has already shown that only a small amount of 3:3'-dichloro-4:4'-dihydroxydiphenyl is obtained by boiling the corresponding diazonium salt with water (*Trans.*, 1903, lxxxiii., 688), and now describes a method for preparing the substance by chlorinating 4:4'-dihydroxydiphenyl.

The following compounds are described:—3:3'-Dichlorodiphenyl, white needles, m. p. 29°, b. p. 298°; 3:4:3':4'-tetrachlorodiphenyl, white needles, m. p. 172°, b. p. 230° under 50 m.m. pressure; 4:4'-dibromo-3:3'-dichlorodiphenyl, white needles, m. p. 176—177°; 4:4'-diiodo-3:3'-dichlorodiphenyl, pale yellow needles, m. p. 162°, b. p. 275° under 10 m.m. pressure; 4:4'-dicyano-3:3'-dichlorodiphenyl, white needles, m. p. 152—153°; 3:3'-dichlorodiphenyl-4:4'-dicarboxylic acid, white needles, m. p. 287—288°. 3-Chloro-4:4'-dihydroxydiphenyl, white needles, m. p. 215°. 3:3':5(?)-Trichloro-4:4'-dihydroxydiphenyl, white needles, m. p. 179°.

170. "Notes on some Natural Colouring Matters." By A. G. PERKIN and E. PHIPPS.

The flowers of the *Prunus spinosa* contain both quercetin and kampherol, whereas in the violet (*Viola odorata*) and white clover (*Trifolium repens*) quercetin alone has been detected. From the Japanese dye-stuff "Fukugi" (botanical origin unknown), a new colouring matter,  $\text{C}_{17}\text{H}_{12}\text{O}_6$ , has been isolated, forming yellow, prismatic needles (m. p. 288—290°), the general properties of which indicate that it is closely allied to luteolin. On bromination, it yields the compound  $\text{C}_{17}\text{H}_{10}\text{O}_6\text{Br}_2$  (yellow needles, m. p. 280°), and, when fused with caustic alkali, phloroglucinol and protocatechuic acid are obtained.

*Morintetraethyl ether*,  $\text{C}_{15}\text{H}_{20}\text{O}_3(\text{OEt})_4$  (yellow, prismatic needles, m. p. 126—128°), closely resembles the corresponding methyl derivative and forms the colourless acetyl compound,  $\text{C}_{15}\text{H}_{18}\text{O}_3(\text{OEt})_4\cdot\text{C}_2\text{H}_3\text{O}$  (needles, m. p. 121—123°). When brominated in the presence of alcohol, myricetin yields a mixture of tetrabromomyricetin and tetrabromomyricetin ethyl ether,  $\text{C}_{15}\text{H}_8\text{Br}_4\text{O}_7\cdot\text{OEt}$  (colourless needles melting completely at 146°).

Cryoscopic determinations of the molecular weight of its acetyl derivative indicate that hesperitin has the formula  $\text{C}_{16}\text{H}_{14}\text{O}_6$ , and not  $\text{C}_{32}\text{H}_{28}\text{O}_{12}$ , as previously suspected by the formation of the alkali salts,  $\text{C}_{32}\text{H}_{27}\text{O}_{12}\text{K}$  and  $\text{C}_{32}\text{H}_{27}\text{O}_{12}\text{Na}$  (*Trans.*, 1898, xxxi., 1031). *Benzoylcurcumin* (yellow needles, m. p. 176—178°) in a similar manner gave results in harmony with Ciamician and Silber's formula,  $\text{C}_{21}\text{H}_{20}\text{O}_6$  (*Ber.*, 1897, xxx., 190), for curcumin.

171. "The Estimation of Methyl Alcohol in Presence of Ethyl Alcohol." By T. E. THORPE and J. HOLMES.

The authors described a method of estimating the amount of methyl alcohol in mixtures of methyl and ethyl alcohols based on the different modes of action of a mixture of potassium dichromate and sulphuric acid on the two alcohols. The method differs from that already described by Dupré, in 1876, in which the amount of acetic acid formed is taken as a measure of the amount of ethyl

alcohol present, in that the amount of carbon dioxide evolved is weighed.

The authors described how their method is applicable to the case of the methylated spirit of commerce, and to the detection and determination of methylated spirit in tinctures and medicinal preparations suspected to contain it.

172. "Separation and Estimation of Silver Cyanide and Silver Chloride." By R. H. A. PLIMMER.

Freshly precipitated silver cyanide, although insoluble in cold dilute nitric acid, readily dissolves in the boiling acid, evolving the theoretical quantity of hydrogen cyanide, so that the gas, when passed into silver nitrate solution, produces an amount of silver cyanide equal in weight to the sample originally employed. In this way, silver cyanide may be quantitatively separated from silver chloride. When the cyanide has been dried at 100°, the hard lumps produced offer a greater resistance to the solvent, and if the boiling is prolonged the acid, on becoming more concentrated, oxidises a small proportion of the hydrogen cyanide.

173. "Estimation of Hydroxyl Radicles." By H. HIBBERT and J. J. SUDBOROUGH.

In attempting to determine hydroxyl groups by Tschugaeff's method (*Ber.*, 1902, xxxv., 3912), the authors have not succeeded in obtaining satisfactory results, owing to the following causes:—1. Moisture gradually penetrates through the india-rubber, even although this is coated with collodion. 2. The vapour pressure of ether varies so enormously with slight alterations of temperature. 3. Grignard's magnesium methyl iodide solution slowly absorbs atmospheric oxygen.

The following method is free from the foregoing objections; dry amyl ether is used as the medium instead of ordinary ether, and the operation is carried out in an ordinary bottle provided with a double bored india-rubber stopper, so that the bottle may be attached to (1) a Lunge's nitrometer filled with dry mercury, (2) a glass tube provided with a stopcock, so that the air in the apparatus may be replaced by dry nitrogen. The amyl ether solution of the hydroxyl compound is placed in the bottle, and a solution of Grignard's magnesium methyl iodide in the same solvent is introduced in a small tube; when a constant temperature has been attained, the two solutions are mixed.

Satisfactory results have been obtained with  $\alpha$ - and  $\beta$ -naphthols, resorcinol, *o*-nitrophenol, acetoxime, deoxybenzoin, and chloral hydrate.

Quinol gives somewhat low results, probably owing to the fact that it is only sparingly soluble in amyl ether.

174. "Diortho-substituted Benzoic Acids. Part V. Formation of Salts from Diortho-substituted Benzoic Acids and Organic Bases." By J. J. SUDBOROUGH and W. ROBERTS.

Since the publication of Part IV. (*Trans.*, 1899, lxxv., 580), E. Fisher has shown (*Ber.*, 1900, xxxiii., 345, 1967) that diortho-substituted tertiary bases of the type of dimethylmesidine are incapable of forming quaternary ammonium salts. The authors have prepared several mono- and diortho-substituted tertiary amines, and find that these are capable of combining with strong diortho-substituted benzoic acids, for example, *s*-trinitrobenzoic and 2:4:6-tribromo-3-aminobenzoic acids, but will not unite with feeble acids like 2:4:6-trimethylbenzoic and *o*-toluic acids.

In addition to the ordinary normal salts (1 mol. acid + 1 mol. base), acid salts of the type (2 mols. acid + 1 mol. base) derived from monobasic acids and monoacidic bases have been obtained. Many of these salts can be analysed by direct titration in alcoholic solution with standard barium hydroxide solution, using phenolphthalein as the indicator.

175. "cis- $\pi$ -Camphanates of *d*- and *l*-Hydrindamines. By F. S. KIPPING.

cis- $\pi$ -Camphanic acid gives with *dl*-hydrindamine two

isomeric salts melting at 173° and 193° respectively, but which have the same specific rotation in aqueous solution (*Trans.*, 1900, lxxvii., 903).

Having recently succeeded in resolving the *dl*-base (*Trans.*, 1903, lxxxiii., 873), the author examined the *cis*- $\pi$ -camphanates of the *d*- and of the *l*-base, partly to prove that the compounds previously described are really partially racemic, and partly to ascertain whether each of the optically active bases gives isomeric salts.

*d*-Hydrindamine *cis*- $\pi$ -camphanate, obtained by combining the acid with the base from pure *d*-hydrindamine *d*-bromocamphorsulphonate, crystallises from water and alcohol in long needles and melts at about 181°; when fractionally crystallised from aqueous alcohol, the final mother liquors yield fractions which melt very indefinitely at 170–175°; the specific rotation of various fractions of this salt, examined in 2 per cent methyl alcoholic solution, varied from  $[\alpha]_D - 7.8^\circ$  to  $-17.8^\circ$ .

*l*-Hydrindamine *cis*- $\pi$ -camphanate, prepared from the base contained in pure *l*-hydrindamine *d*-bromocamphorsulphonate, closely resembles the salt of the *d*-base, but the original preparation melts indefinitely at 186–191°; when crystallised fractionally from aqueous alcohol, it yields portions melting as low as 180–184°. The specific rotation of fractions of this salt, determined in 2 per cent methyl alcoholic solutions, varied from  $[\alpha]_D - 6^\circ$  to  $+7^\circ$ .

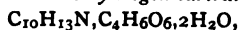
These observations seem to justify the conclusion that each base gives two isomeric salts, which, when the *dl*-base is used, combine in pairs to form the two partially racemic compounds previously described. The whole of the *cis*- $\pi$ -camphanic acid used in these and in previous experiments was collected and crystallised fractionally from alcohol: the first and last fractions crystallised in identical forms, namely, characteristic hexagonal plates (*Trans.*, 1896, lxix., 943), melted simultaneously, and had the same specific rotation in alcoholic solution.

176. "Resolution of *dl*-Methylhydrindamine. By G. TATTERSALL.

In resolving *dl*-methylhydrindamine into its enantiomorphously related components by the use of *d*-bromocamphorsulphonic acid (*Trans.*, 1903, lxxxiii., 918), the yield of salts of the *d*- and *l*-bases was always small, and the author, at Dr. Kipping's suggestion, attempted to find a more satisfactory process for the resolution of this *dl*-base.

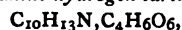
Experiments then showed that *d*-tartaric acid, used so as to form the hydrogen tartrates of the base, effected an excellent separation of the *d*- and *l*-bases by crystallising the salt at the ordinary temperature. The most sparingly soluble fractions contained the hydrogen tartrate of the *d*-base. The purification of the bases was effected by converting the first fractions, containing chiefly the *d*-base (about one third of the whole), and the last fractions separately into the *d*-bromocamphorsulphonates and finally fractionating from water.

*d*-Methylhydrindamine hydrogen tartrate,—



crystallises from water in long, glassy prisms; it melts at 153–155° with slight decomposition. In aqueous solution, it has a specific rotation  $[\alpha]_D +33^\circ$  and molecular rotation  $[M]_D +99^\circ$ .

*l*-Methylhydrindamine hydrogen tartrate,—



crystallises from water in aggregates of small, glassy, prisms; it melts at 197° with slight decomposition. In aqueous solution, its specific rotation is  $[\alpha]_D -5^\circ$  and its molecular rotation  $[M]_D -14.8^\circ$ .

177. "Isomeric Salts of *d*- and *l*-Methylhydrindamines with *d*-Chlorocamphorsulphonic Acid." By G. TATTERSALL.

In a recent communication (Tattersall and Kipping, *Trans.*, 1903, lxxxiii., 918), the formation of isomeric salts by the combination of *d*- and *l*-methylhydrindamines with *d*-bromocamphorsulphonic acid has been described, and

similar experiments have now been made with the same bases and *d*-chlorocamphorsulphonic acid.

The salt of each base was made from carefully purified material and subjected to prolonged fractional crystallisation; the end fractions were then examined. In the cases of both the *d*- and *l*-bases, the melting-points of the successive fractions gradually fell from the first to the last fractions, this behaviour being also observed with the *d*-bromocamphorsulphonate.

The chlorocamphorsulphonates differ, however, from the bromocamphorsulphonates in their optical behaviour. With the former acid, the specific rotations of the last fractions are higher than those of the first fractions, whereas with the latter acid the opposite was the case. The statement is true for both aqueous and chloroform solutions.

The results obtained are summarised in the following table:

*l*-Methylhydrindamine *d*-chlorocamphorsulphonate.

|                | m. p.    | In chloroform. | In water. |
|----------------|----------|----------------|-----------|
| First fraction | 239°     | +3.3°          | +34.2°    |
| Last "         | 231—233° | 8.0            | 35.8      |

*d*-Methylhydrindamine *d*-chlorocamphorsulphonate.

|                | m. p.    | In chloroform. | In water. |
|----------------|----------|----------------|-----------|
| First fraction | 247°     | +56°           | +60°      |
| Last "         | 225—230° | 63             | 63        |

Both salts crystallise in anhydrous needles.

Similar results were obtained with hydrindamine *d*-chlorocamphorsulphonates (Kipping, *Trans.*, 1903, lxxxiii., 902), but in this case it was found impossible to isolate the isomeride of higher specific rotation.

178. "The Four Optically Isomeric *l*-Menthylamines and their Salts." By F. TUTIN and F. S. KIPPING.

The principal object of this research was to ascertain whether *l*-menthylamine would give, with *d*-bromocamphorsulphonic acid, isomeric salts analogous to those obtained by combining this acid with the optically active hydrindamines (*Trans.*, 1903, lxxxiii., 873) and methylhydrindamines (*loc. cit.*, p. 918): for this purpose, it was first necessary to obtain the *l*-menthylamine free from optical isomerides. An examination of the menthylamine prepared by reducing the oxime of "*l*-menthone" showed that the crude basic product was a mixture of four optically isomeric active bases, and that the menthylamine obtained by heating "*l*-menthone" with ammonium formate was likewise composed of these four isomerides.

The explanation of these facts is afforded by a consideration of the optical configuration of *l*-menthone—a ketone which readily undergoes partial racemisation—and also of the reactions by means of which it is transformed into the base.

Since the four isomerides in question are derivatives of *l*-menthone (—) and of *iso-l*-menthone (+), they are distinguished as *l*-menthylamine, neo-*l*-menthylamine, *iso-l*-menthylamine (*d*-menthylamine in the literature), and *isoneo-l*-menthylamine respectively.

*l*-Methylamine combines with *d*-bromocamphorsulphonic acid, giving a mixture of isomeric salts. One of these compounds can be obtained in a pure condition by repeated fractional crystallisation from water; it melts at about 226° and has a specific rotation  $[\alpha]_D^{20} +43.7^\circ$  in aqueous, and  $[\alpha]_D^{20} +48.7^\circ$  in chloroform solution. The isomeric salt cannot be obtained in a pure condition, but its presence is conclusively proved by the fact that the more readily soluble fractions melt at about 221° and have a specific rotation  $[\alpha]_D^{20} +38.8^\circ$  in aqueous, and  $[\alpha]_D^{20} +42.8^\circ$  in chloroform solution.

For the separation of the four isomeric *l*-menthylamines in the crude base, their hydrochlorides, *d*-bromocamphorsulphonates, and *d*-camphorsulphonates were fractionally crystallised, and also their formyl derivatives; *l*-menthylamine is thus isolated without difficulty, but for the separation of the other three isomerides, fractional crystallisation

of their benzoyl derivatives was found to give the best results.

The following compounds are described:—

|                                  | M.p.                     | $[\alpha]_D^{20}$ |
|----------------------------------|--------------------------|-------------------|
| <i>d</i> -Bromocamphorsulphonate | <i>l</i> -Base .. ..     | 226° +43.2°       |
|                                  | neo- <i>l</i> -Base ..   | 70—75° —          |
|                                  | <i>iso-l</i> -Base ..    | 166 +65           |
|                                  | <i>isoneo-l</i> -Base .. | —                 |
| <i>d</i> -Camphorsulphonate ..   | <i>l</i> -Base .. ..     | 158 — 5.3         |
|                                  | neo- <i>l</i> -Base ..   | 188 +11.8         |
|                                  | <i>iso-l</i> -Base ..    | 177 +21.7         |
|                                  | <i>isoneo-l</i> -Base .. | —                 |
| Benzoyl derivative .. ..         | <i>l</i> -Base .. ..     | 156 — 61.9        |
|                                  | neo- <i>l</i> -Base ..   | 128 — 17.4        |
|                                  | <i>iso-l</i> -Base ..    | 121 +22.7         |
|                                  | <i>isoneo-l</i> -Base .. | 104 — 3.8         |

The specific rotations of the salts were determined in aqueous solutions, those of the benzoyl derivatives in chloroform.

179. "Preparation of the Tetra-Alkyl Derivatives of Stannimethane." By W. J. POPE and S. J. PEACHEY.

The preparation of the mixed tetra-alkylstannimethanes by the action of a zinc alkyl upon a trialkylstannimethyl iodide is difficult to carry out owing to the occurrence of secondary reactions, which greatly diminish the yield of the desired product. The authors have found that the magnesium alkyl iodides, bromides, and chlorides prepared by the method given by Grignard react vigorously with stannic chloride or with the mono-, di-, and tri-alkylstannimethane halogen derivatives, giving rise to the corresponding tetra-alkylstannimethanes. After treating the product with water, the tetra-alkyl derivative is readily separated in a state of purity by fractional distillation of the ethereal solution.

Tetraethylstannimethane,  $\text{SnEt}_4$ , is prepared by adding stannic chloride (1 mol.), dissolved in light petroleum, to the ethereal solution of magnesium ethyl bromide (4.5 mols.), washing with dilute acetic acid and distilling the ethereal solution; it boils at 180—181° under 758 m.m. pressure.

Dimethyldiethylstannimethane,  $\text{SnMe}_2\text{Et}_2$ , may be prepared either by the action of magnesium methyl iodide on diethylstannimethylene chloride, bromide, or iodide, or by that of magnesium ethyl bromide on dimethylstannimethylene chloride, bromide, or iodide in ethereal solution. After treating with water and distilling the ethereal solution, the substance is obtained as a colourless liquid identical with that prepared by Frankland's method.

Trimethylethylstannimethane,  $\text{SnMe}_3\text{Et}$ , is obtained by treating trimethylstannimethyl bromide or iodide with the calculated quantity of magnesium ethyl bromide in ethereal solution, washing with dilute acetic acid, and fractionally distilling the ethereal solution; it boils at 107—108° under 758 m.m. pressure.

Trimethylpropylstannimethane,  $\text{SnMe}_3\text{Pr}$ , is similarly prepared by the action of magnesium propyl bromide or iodide on trimethylstannimethyl bromide and forms a colourless liquid boiling at 129° under 764 m.m. pressure. Triethylpropylstannimethane,  $\text{SnEt}_3\text{Pr}$ , from triethylstannimethyl bromide, boils at 195° under 764 m.m. pressure.

Dimethylethylpropylstannimethane,  $\text{SnMe}_2\text{EtPr}$ , prepared by the action of propyl magnesium bromide or iodide on dimethylethylstannimethyl bromide, boils at 153° under 762 m.m. pressure.

Methylethyldipropylstannimethane,  $\text{SnMeEtPr}_2$ , obtained from methylethylstannimethylene iodide and magnesium propyl bromide, is a colourless liquid boiling at 183—184° under 758 m.m. pressure.

Tetraphenylstannimethane,  $\text{SnPh}_4$ , is conveniently prepared by the action of stannic chloride on magnesium phenyl bromide.

The further study of the mixed alkylstannimethanes, prepared by aid of the above reaction, is being continued, together with the examination of the action of the magnesium alkyl salts on silicon tetrachloride.

180. "Optically Active Esters of  $\beta$ -Ketonic and  $\beta$ -Aldehydic Acids. Part IV. Condensation of Aldehydes with Menthyl Acetoacetate." By A. C. O. HANN and A. LAPWORTH.

Menthyl acetoacetate condenses readily with aldehydes in presence of bases, and the following products have been obtained.

*Dimenthyl ethylidenebisacetoacetate*,—



produced when acetaldehyde is used, forms fine needles melting at  $194-196^\circ$  and has  $[\alpha]_D - 24.9^\circ$  in benzene, a very slight mutarotation being noticed.

*Menthyl n-propylidenebisacetoacetate*,—



is formed at the ordinary temperature, crystallises in glistening, elongated plates or needles, melts at  $84-88^\circ$ , and has  $[\alpha]_D - 34.9^\circ$ . It condenses with menthyl acetoacetate to form *dimenthyl n-propylidenebisacetoacetate*,  $\text{CHEt}(\text{CHAc} \cdot \text{CO}_2 \cdot \text{C}_{10}\text{H}_{19})_2$ , which melts at  $201-207^\circ$  and has  $[\alpha]_D - 26.9^\circ$ .

*Dimenthyl n-butylidenebisacetoacetate*,—



melts at about  $184^\circ$  and has  $[\alpha]_D - 16.8^\circ$ . *Dimenthyl isobutylidenebisacetoacetate*,  $\text{CHPr}^i(\text{CHAc} \cdot \text{CO}_2 \cdot \text{C}_{10}\text{H}_{19})_2$ , melts at  $193-202^\circ$ ; in benzene solution, it has  $[\alpha]_D - 42.6^\circ$ , changing slowly to  $[\alpha]_D - 46.0^\circ$ .

*Menthyl benzylidenebisacetoacetate*,—



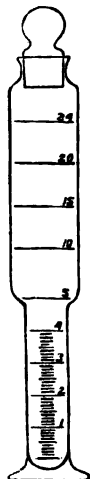
crystallises in well-formed, glistening plates melting at  $133-134^\circ$ ; in benzene solution,  $[\alpha]_D - 10.0^\circ$ . With menthyl acetoacetate, it yields *menthyl benzylidenebisacetoacetate*,  $\text{CHPh}(\text{CHAc} \cdot \text{CO}_2 \cdot \text{C}_{10}\text{H}_{19})_2$ , which melts at  $203-206^\circ$  and has  $[\alpha]_D - 30.4^\circ$ .

The formula usually ascribed to the monoalkylidene derivatives of  $\beta$ -ketonic esters does not readily account for the abnormally low values of their rotatory powers.

The statement that tertiary bases are not effective in bringing about the condensation of aldehydes with  $\beta$ -ketonic esters is incorrect; the very feeble tertiary bases, pyridine, quinoline, or dimethylaniline, are useless, but trimethylamine and tripropylamine may be employed, although these are not so rapid in their action as the still more powerful secondary bases, such as diethylamine or piperidine.

181. "Estimation of the Adulterant in Citronella Oil." By M. K. BAMBER.

A mixture of 2 c.c. of pure coconut oil free from acid



and 2 c.c. of the citronella oil under examination is shaken for one minute with 20 c.c. of 83 per cent of alcohol (sp. gr.

0.8273 at  $30^\circ$ ) in the graduated tube (see Fig.), this vessel being then rotated in a centrifugal machine for 0.5 to 1 minute. The volume of coconut oil, which now contains the impurity originally present in the citronella oil, is ascertained, and this reading minus 2 c.c. represents the adulterant. For example, 2.45 c.c. of residual oil represent 0.45 c.c. of impurity in the 2 c.c. of citronella oil or an adulteration of 22.5 per cent. A standard oil should be tested occasionally against the unknown samples in order to eliminate errors arising from the use of alcohol of different strengths.

In this way, the adulterant is separated and estimated in three or four minutes, the test being conducted at  $29-30^\circ$ .

## NOTICES OF BOOKS

*Elementary Practical Chemistry*. Part I., General Chemistry. Part II., Analytical Chemistry, Qualitative and Quantitative. By FRANK CLOWES, D.Sc. (Lond.), and J. B. COLEMAN, A.R.C.Sc. Fourth Edition. London: J. and A. Churchill. 1903. Pp. 212 and 198.

THE present edition has been published in two parts. Part I. has been arranged to suit the requirements of the general science student, and it is hoped that it will be found convenient to students of this class; students of pure and applied chemistry who require careful training in analytical methods will find a complete course of qualitative analysis, including a few examples of quantitative methods in Part II. Most students will require both parts if they really intend to make a serious study of chemistry, and if their chemical future is not bounded by the inevitable exam.; still, we are of the opinion that this division of the book is a wise one, as the two volumes separately are more handy in form than the one larger one.

It is a pity, however, that the two volumes have not been better matched; the paper used for Part II. is quite different to that used in Part I., and even the lettering on the backs is not in the same style.

Part I. commences with a description of the metric system, and gradually lays the principles of chemistry before the student by means of a selected series of experiments. Many quantitative experiments are given, and this part concludes with a selection of examples to test the progress of the student.

Part II. begins with descriptions of various ordinary chemical operations, such as ignition, precipitation, and filtration. Then follow descriptions of the reactions and methods of detection of the common metals and acids.

Quantitative analysis, both gravimetric and volumetric, is next in order; the volumetric methods described include acidimetry and alkalimetry, and estimations by means of oxidation, reduction, and precipitation. The gravimetric estimations are the well-known ones, and are combined with the usual group reagents for the separation of the metals.

The general arrangement of these two volumes is good, as is also the printing, while each volume is provided with an adequate table of contents and an index.

*Year-Book of Pharmacy*. Edited by J. O. BRAITHWAITE. London: J. and A. Churchill. 1903. Pp. 659.

THIS volume comprises abstracts of papers relating to materia medica, pharmacy, and chemistry, contributed to British and foreign journals from July 1st, 1902, to June 30th, 1903, with the Transactions of the British Pharmaceutical Conference at the fortieth annual meeting, held in Bristol, July, 1903. The compilation of such a volume is by no means a light undertaking, and the editors are to be congratulated on the satisfactory result of their labours, which will no doubt be of service to all pharmaceutical chemists.



*Annual for the Year 1904.* ("Annuaire pour l'An 1904.")  
Published by the Bureau des Longitudes. Paris:  
Gautier-Villars. Pp. 732.

CERTAIN new arrangements are here inaugurated in the compilation of this annual. For instance, detailed tables are given relative to physical and chemical science, while the geographical and statistical tables have been omitted.

In the astronomical portion, the data relative to calendars, and more especially ancient calendars, have received more attention, and a complete table of the elements of the small planets has been inserted. Among the matters that have been omitted are the calculation of altitudes from the height of the barometer, stellar parallaxes, double stars, &c.

The physical portion contains many additions, such as the magnetic elements at various points on the earth's surface; the dilatation of various liquids; the vapour tension of various liquids, mercury in particular; a table of wave lengths, by M. de Gramont; the solubility of various substances in water, &c., and the chemical portion has been completed by the addition of a table of the principal alloys.

*Dictionary of Hygiene.* B. C. T. KINGZETT, F.I.C., and D. HOMFRAY, B.Sc. Second Edition. London: Ballière, Tindall, and Cox, 1904. Pp. 112.

IN these pages the authors have given concise information respecting most of the subjects comprehended in the theory and practice of hygiene, so far as they can be dealt with in pocket-book form. In this edition several fresh articles have been introduced, and some of the subjects dealt with in the previous edition have been treated more fully. The book is handy in form, and as it measures only 3 x 5 inches, it will easily go in the pocket, where it is always ready for reference.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

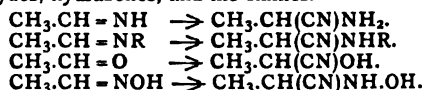
*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxvii., No. 23, December 7, 1903.

**A New Method of Calculating Heats of Combustion.**—P. Lemoult.—In a previous communication the author showed that the heat of combustion of carbides and their oxygen derivatives can be calculated from that of the elementary groups  $(C_3 \equiv C_3)$ ,  $-(C_2 = C_2)$ ,  $\dots (C-H)$ , and of the functional groups. This method led him to apply two series of formulæ to extend his research:—(1)  $C_1 = 157n + A_1$ ; (2)  $C_2 = 463m + A_2 \equiv 115.75n + A_2$ . The first for acyclic compounds, the second for cyclic compounds ( $n$  being the number of atoms of C,  $m$  the number of the order of the generating cyclic carbide).

**Researches on Azoic Compounds, New Method of Formation of Indazylic Derivatives.**—P. Freundler.—Investigations on azoic compounds possessing an alcoholic or ortho-substituted ether oxide function show the facility with which the indazylic radicle is produced; *o*-azobenzoic and *o*-hydrazobenzoic acetals furnish a very striking example. The transformation of these compounds into indazols can be effected at a comparatively low temperature, and under the influence of agents which are not very energetic. Further, this implies a previous modification of the functional groups which cannot usually be effected during the conditions under which the author works.

**Action of Hydrocyanic Acid on Ammonium Aldehyde and Analogous Combinations.**—Marcel Delépine.—The action of hydrocyanic acid on the am-

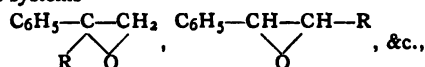
monium aldehydates constitutes one of the methods of synthesis of the  $\alpha$ -amino-acids. The transformations may be represented generally by various equations according to different experimenters, and the author now shows that the classic equations which show the elements of water intervening and expressing a theoretical yield of aminonitrite ought to be modified. The most simple modification is first to assume that the hydrocyanic acid fixes itself on to the double liaisons of the imines as is the case with the aldehydes, hydrazones, and the oximes.



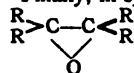
**New Action of Hydroxylamine.**—L. J. Simon.—When a few drops of a very dilute solution of sodium nitroprussiate and a small excess of soda or potash are added to a dilute solution of a salt of hydroxylamine, and the liquid is gradually raised to boiling-point, it changes its colour from yellow, first to an orange-red, and finally takes a beautiful cerise red colour. During this process nitrogen and nitrous oxide gas are evolved. This reaction can often be used with great advantage for the identification of hydroxylamine.

**New Method of Preparation of Aldehydes.**—L. Bouveault.

**Phenyl Migration.**—Marc Tiffeneau.—The author makes a series of experiments on phenyl migration, and comes to the conclusion that in the passage from the unstable form of ethylene oxide to the corresponding stable form, aldehyde or ketone, the phenyl radicle is more mobile than the hydrogen, and this in its turn more mobile than the alkyl radicle (ethyl, amyl, or benzyl). In the various systems—



it is always the phenyl which migrates, whilst in the corresponding systems where the  $C_6H_5$  is replaced by an alcoholic radicle, it is always the hydrogen and not the alkyl which migrates. Finally, in systems such as—



showing no free hydrogen atom, it is the phenyl which tends to migrate.

**Ethers of Isopyromucic Acid.**—G. Chavanne.—Methylic and ethylic ethers of isopyromucic acid can not be obtained by any of the usual methods. The author succeeds, however, in isolating them by the use of dimethylic and diethylic sulphates. Their stability towards water and dilute acids places them rather amongst the ethers of phenols than amongst the ether salts.

**Ethyl Alcohol Hydrates.**—E. Varenne and L. Godefroy.—The hydrate of alcohol,  $C_2H_5.OH + 3H_2O$ , has been known for some time. This corresponds to 52.3 vols. of alcohol mixed with 47.7 vols. of water at 15°, and gives the maximum of contraction. In order to investigate various mixtures and compounds of alcohol and water the authors use a special apparatus called a chrono-stiloscope, of which they describes the construction and use.

*Bulletin de la Société Chimique de Paris.*

Series 3, Vol. xxix., No. 10.

This number contains no chemical matter that has not been already noticed in our columns.

No. 11.

**Application of the Law of Phases to the Examination of the Distillation of Pine-resin.**—M. Vèzes.—A long paper not suitable for abstraction.



**Silicic Acid and the Alkaline and Alkaline-earthly Silicates.**—E. H. Kanter.—The experiments and the research described in this paper show that, contrary to general belief, there is a much larger number of soluble silicates, though they are difficultly soluble. The research also explains a number of contradictions met with in papers dealing with the silicates, and it opens up a new field for experiment.

**The Electrolytic Reduction of Chlorate of Potassium.**—D. Tommasi.—Already noticed.

**Preparation of Barium.**—M. Guntz.—Already noticed.

**The Sub-salts of Barium.**—M. Guntz.—Already noticed.

**Action of Ammonia Gas at a Low Temperature on Barium (Barium-ammonium).**—M. Mentrel.—A long paper which cannot be well abstracted without the accompanying diagrams.

**The Combination of the Hexatomic Alcohols with the Mononitrobenzaldehydes.**—Adolphe Simonet.—The author was desirous of seeing what the behaviour of certain high polyatomic alcohols would be when brought into reaction with the derivatives of benzaldehyde, such as the nitrated derivatives, for example. For this purpose he used mannite, sorbite, and dulcitol, and he observed that the compounds obtained differed according to the nitrobenzaldehyde used. Thus mannite gave a trimetanitrobenzalmannite with meta-nitrobenzaldehyde, and a monoparanitrobenzalmannite with para-nitrobenzaldehyde. Sorbite and dulcitol gave similar bodies when treated with the same nitrobenzaldehydes. No results were obtained with ortho-nitrobenzaldehyde. The method of obtaining the above compounds is described for each, together with their analysis.

**The Saponification of the Nitric Ethers.**—Leo Vignon and I. Bay.—Already noticed.

**Constitution of the Nitrocelluloses.**—Leo Vignon.—Already noticed.

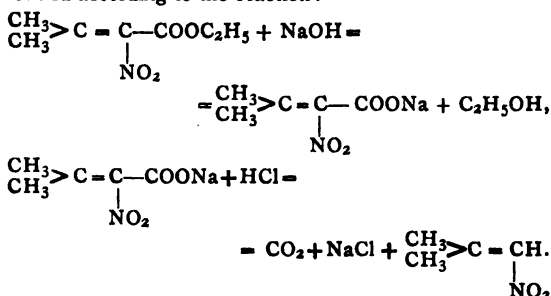
**Nitrated Cellulose.**—Leo Vignon.—Already noticed.

**Soluble Cellulose.**—Leo Vignon.—Already noticed.

**The Influence of Copper on the Silvering of Glass.**—Leo Vignon.—The author has observed that very small quantities of copper, such as that present in distilled water made with a copper retort, have an influence on the silvering of glass in the manufacture of looking-glasses. The results of his experiments show that the presence of a very small quantity of copper (0.8 m.grm. per litre) facilitates the operation by lowering the temperature at which the mirror is formed. A higher proportion of copper alters the colour of the mirror, and may even prevent its formation.

**Reduction of  $\omega$ -Nitrostyrolene.**—L. Bouveault and A. Wahl.—The reduction of this substance is effected by means of aluminium amalgam, or with powdered zinc and acetic acid. In the first case the operation is conducted as with nitrodimethylacrylate of ethyl or nitro-isobutylene, but the best results were obtained with zinc powder. Into a flask fitted with a good condenser we put 30 grms. of nitrostyrolene and 200 to 300 c.c. of ordinary ether; 60 grms. of zinc powder are added; then slowly a few c.c. of acetic acid at 75 per cent. The flask gradually becomes hot and a lively reaction is set up, which is shown by the boiling of the ether. Ninety grms. more of acetic acid at 75 per cent are gradually added. When the reaction is completed, the zinc and the acetate of zinc are separated by draining, or by adding water and separating the ether by decantation. The ether is washed several times, and evaporated to half its volume and cooled. Small crystalline flakes separate out; these are removed and the filtrate gradually deposits long needles, which are re-crystallised in a mixture of ether and petroleum ether; they then melt at 103°. The analysis of this body gives the formula  $C_{10}H_9NO$ , which is that of the oxime of phenylacetic aldehyde.

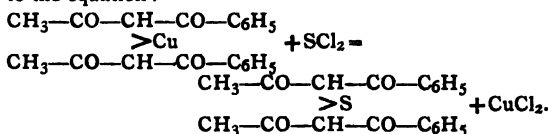
**Nitro-isobutylene.**—L. Bouveault and A. Wahl.—To prepare nitro-isobutylene it is best to proceed in the following manner:—Heat 70 grms. of nitrodimethylacrylate of ethyl to 50° on the water-bath with 200 to 250 c.c. of caustic soda at 20 per cent, stirring frequently. The product dissolves rapidly, and finally we obtain a clear yellow coloured liquid. On acidulating, a slightly coloured oil is precipitated and carbonic acid is given off in abundance; the oil is collected in ether, and the ether washed with carbonate of soda, then with water, and distilled. The nitro-isobutylene passes over at 80° under 40 m.m. pressure, after which a higher portion comes over at 106° under 10 m.m. pressure; this consists of a mixture of nitrodimethylacrylate of ethyl  $\alpha$  and  $\beta$ , partially soluble in cold soda and completely so when heated. Nitro-isobutylene is formed according to the reaction:—



It is a very mobile, pale yellow liquid, having an extremely irritating odour, its density at 0° = 1.052.

**Preparation and Reduction of the Homologues of Nitrostyrolene.**—L. Bouveault and A. Wahl.—A long paper not suitable for abstraction.

**Thiobenzoylacetone.**—V. Vaillant.—Thiobenzoylacetone is coloured slightly yellow, and is formed according to the equation:—



It is insoluble in water, and barely soluble in alcohol even when boiling, but it dissolves easily in most of the organic solvents. It forms salts with sodium, copper, iron, and ammonium.

**Some Compounds by means of which the Constitution of Bismuthogallic Acid may be Determined.**—Paul Thibault.—In a previous paper the author has shown that the supposed basic gallate of bismuth is capable of forming alkaline compounds, and consequently is not a salt of bismuth. On account of its acid character he calls it bismuthogallic acid. As a result of further researches he describes certain compounds which prove distinctly the existence of a free acid function in the bismuthogallic compound, which proves that the bismuth is fixed to the phenolic radical of the gallic acid.

**Oxidation of Urinary Indoxyl into Indigotic Colours.**—L. Maillard.—A long paper not suitable for abstraction.

**Estimation of Formic Aldehyde in the Air.**—G. Romyn and J. A. Voorthuis.—Principally a review of the known methods of making this estimation.

**Some Properties of Colloidal Silver.**—A. Chassevant and S. Posternak.—Already inserted in full.

**Elimination and Distribution in the Organism of Medicinal Arsenic in the Form of Methyl Arsenate of Soda.**—A. Mouneyrat.—The author shows that whatever the dose of methyl arsenate of soda absorbed, there is a maximum elimination in the first four or five hours after the absorption; then from this moment, its excretion, for

the same lapse of time, diminishes progressively. After twenty-four hours, about three-fifths of the substance is eliminated, but the whole is not eliminated until the thirty-second day.

**Influence of the Chemical Form under which an Element is Presented to the Organism, on the Rapidity of its Passage into the Blood.**—A. Mouneyrat.—As a result of experiments made with arsenic on dogs, the author finds that the blood of dogs that received the arsenic in the mineral form (arsenate and arsenite), contained more arsenic than that of dogs that received the same metalloid in the organic form.

**The Solubility of Hard Copals.**—Ch. Coffignier.—Experiments show that Zanzibar copal is more insoluble than that from Madagascar, no matter what solvents were used. The solvents tried were ethylic, methylic, and amylc alcohols, sulphuric ether, chloroform, acetone, benzene, aniline, acetate of amyl, and others. The actual results are all given in tabular form.

### MISCELLANEOUS.

**South - Western Polytechnic, Chelsea.**—The Governing Body of this Institute have accepted with very great regret the resignation of the Principal, Mr. Herbert Tomlinson, F.R.S. At their meeting held December 16th, 1903, the following resolution was passed:—"That the Governing Body hereby desire to record their cordial appreciation of the admirable work that Mr. Tomlinson, as the first Principal, has accomplished in organising and developing the Institute in all its branches."

**Royal Institution.**—On Tuesday next, January 12, at 5 o'clock, Prof. L. C. Miall will commence a course of six lectures at the Royal Institution on "The Development and Transformations of Animals"; on Thursday, January 14, at the same hour, Mr. G. R. M. Murray delivers the first of three lectures on "The Flora of the Ocean"; and on Saturday, January 16, at 3 o'clock, Mr. J. A. Fuller Maitland begins a course of three lectures on "British Folk Song" (with Vocal Illustration). The Friday Evening Discourse on January 15 will be delivered by the Rt. Hon. Lord Rayleigh, his subject being "Shadows"; on January 22 by the Rev. W. Sidgreaves on "Spectroscopic Studies of Astrophysical Problems at Stonyhurst College Observatory"; and on January 29 by Mr. D. G. Hogarth on "The Marshes of the Nile Delta."

**Iodometric Estimation of Thallium by Precipitation in the Form of Chromate.**—E. Rupp.—The volumetric estimation of thallium proposed by Messrs. Browning and Hutchins gives only approximate figures (*Am. Chem. Soc.*, vol. i., p. 35). The difficulty consists in the easy solubility of the chromate of thallium in water (100 parts of water dissolves 0.03 grm. of the salt at 60°). However, we can obtain exact results by proceeding as with bismuth. The following is the method recommended by Rupp for making the estimation:—Take 10 or 20 c.c. of a 5 per cent solution of chromate of potassium, in which the proportion of  $\text{CrO}_3$  is known exactly, and place them in a 50 or 100 c.c. beaker, with an equal quantity of water, and 1 or 2 grms. of carbonate of lime. To this solution we add the solution of the salt of thallium, which should be neutral, and should not contain more than 0.1 grm. of thallium. We then make the liquid up to a known volume, leave it to stand for thirty minutes, throw on a filter, and estimate the excess of  $\text{CrO}_3$  on an aliquot part of the filtrate (about 20 to 50 c.c.). To effect this, dilute to 100 c.c., add 10 or 15 c.c. of 25 per cent hydrochloric acid, then 1 grm. of iodide of potassium; let stand for five minutes, and titrate with hyposulphite. By making the same estimation on the original chromate solution, we obtain, by difference, the chromic acid utilised for the precipitation of the thallium. The number of c.c. of decinormal hyposulphite corresponding to this chromic acid,  $\times 0.01362$ , gives the amount of thallium.—*Zeit. Anorg. Chem.*, vol. xxxiii., p. 156.

### MEETINGS FOR THE WEEK.

- TUESDAY, 12th.**—Royal Institution, 5. "Development and Transformations of Animals," by Prof. L. C. Miall, F.R.S.  
**WEDNESDAY, 13th.**—Society of Arts, 8 (Juvenile Lectures). "Navigation of the Air," by Eric Stuart Bruce, M.A.  
**THURSDAY, 14th.**—Society of Arts, 4.30. "The Presidency of Bombay," by Sir William Lee-Warner, K.C.S.I.  
Royal Institution, 5. "The Flora of the Ocean," by G. R. M. Murray, F.R.S.  
**FRIDAY, 15th.**—Royal Institution, 9. "Shadows," by The Rt. Hon. Lord Rayleigh, O.M., F.R.S., &c.  
**SATURDAY, 16th.**—Royal Institution, 3. "British Folk Song" (with Vocal Illustrations), by J. A. Fuller Maitland, M.A.

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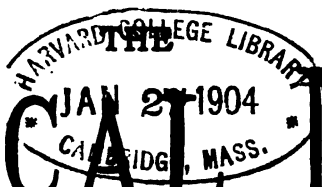
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### INDEX NUMBER,

CONTAINING  
TITLE-PAGE, INDEX, &c., TO VOLUME LXXXVIII.

#### CONTENTS.

| ARTICLES:—                                                                                                                                      | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------------|------|
| On the Gaseous Carbon Monosulphide described by Thomsen, by Norman Smith, M.Sc. ....                                                            | 25   |
| Note on the connection between Negative Electricity and the Valence of Atoms, by Geoffrey Martin, B.Sc. (Lond.) ....                            | 25   |
| Precipitation of some Alkaloids by Nitrate of Uranium—Reaction of Morphine, by J. Aloy ....                                                     | 26   |
| The Use of Binoxide of Lead in Analysis, by M. St. Bogdan ....                                                                                  | 26   |
| Behaviour of Phenolphthalein in the presence of Bicarbonates and of Alkaline Carbonates, by M. Girard ....                                      | 27   |
| The Behaviour of Cerium, Lanthanum, Didymium, Neodymium, Praseodymium, Thorium, and Zirconium towards Organic Bases, by Burt Laws Hartwell .... | 27   |
| The Determination of Benzene in Illuminating Gas, by L. M. Dennis and J. G. O'Neill ....                                                        | 29   |
| The Thermo-chemistry of the Theory of Electrolytic Dissociation, by Joseph W. Richards ....                                                     | 31   |
| A Proposed Method of Testing Wood Treated to Resist Fire, by Chas. F. McKenna ....                                                              | 32   |
| CORRESPONDENCE.—Radium ....                                                                                                                     | 33   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES. ....                                                                                                     | 33   |

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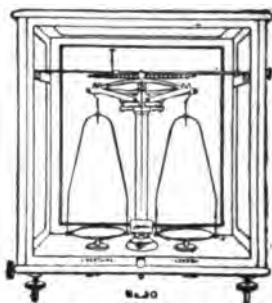
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2303.

## ON THE GASEOUS CARBON MONOSULPHIDE DESCRIBED BY THOMSEN.

By NORMAN SMITH, M.Sc.,  
Demonstrator in Chemistry, The Owens College, Manchester.

WITHIN recent years a number of attempts have been made to prepare carbon monosulphide. In 1895 Deninger (*Journ. Pr. Chem.* [2], li., 346) stated that he had obtained it by the action of sodium on a mixture of carbon disulphide and aniline, but it was shown by Russell and the author (*Journ. Chem. Soc.*, 1902, lxxxi., 1528) that the gases produced were invariably mixtures of already known substances. Last year, Julius Thomsen (*Zeit. f. Anorgan. Chem.*, 1903, xxxiv., 187), by passing a mixture of carbon disulphide and nitrogen over copper heated to dull redness, observed an increase in volume and obtained a gas which, as only nitrogen carbon disulphide and copper were originally present, he concluded was probably carbon monosulphide. On treatment with aqueous potash a small part of the gas was absorbed, due he considered to a small amount of carbon disulphide left in the gas. The residue on sparking with oxygen over aqueous potash, consumed an amount of oxygen approximately corresponding to the amount required by a substance with the formula CS. The small difference between the quantity actually used and the theoretical amount was explained by the formation of some sulphur trioxide.

Since the publication of Thomsen's paper, I have been engaged in repeating the experiments, and in carrying out a large number of somewhat tedious analyses to determine if carbon monosulphide is formed by the above method. I find that the results obtained by Thomsen can be entirely explained without assuming the formation of carbon monosulphide, for during the reaction there are invariably formed oxides of carbon, a mixture of which with carbon disulphide would give the same analytical results as were obtained by him.

Stock and K  chler (*Ber.*, 1903, xxxvi., 4336) have published a paper on the same subject since my experiments were performed. They have repeated Thomsen's experiments and tried to isolate the gas CS by condensation with liquid air, but could not obtain any evidence of a sulphur gas besides carbon disulphide. Further, they have shown that some carbon dioxide is always present, due to the fact that the copper cannot be got perfectly free from oxide.

As the question of the formation of carbon monosulphide has been thrown in doubt by these investigators, I thought it would be of interest to communicate my results, thus independently confirming the conclusions arrived at by Stock and K  chler. I do not, however, consider it necessary under the circumstances to give details of the many analyses performed.

### Experimental.

At first Thomsen's experiments were repeated in the manner described by him, and the gas obtained treated with aqueous potash. The rapid absorption of a portion of the gas which took place was undoubtedly due to carbon dioxide, for carbon disulphide is only very slowly removed by aqueous potash. The residual gas was then exploded with excess of oxygen, and the sulphur dioxide and carbon dioxide produced, as well as the resulting contraction and amount of oxygen used, were determined. From these data one can calculate the amount of sulphur trioxide which is always formed in the explosion (see Russell, *Journ. Chem. Soc.*, 1900, lxxvii., 352). Varying

results were obtained with different samples of gas. In some the volumes of carbon dioxide and sulphur dioxide respectively were approximately equal, indicating a composition CS, but in other experiments, and particularly after the gas had been previously cooled in order to condense most of the carbon disulphide, the volume of carbon dioxide was larger than that of the sulphur dioxide. This led one to suspect the presence of carbon monoxide, which was further indicated by the fact that a part of the original gas was absorbed by acid cuprous chloride. Samples of the gas were then prepared and treated first with alcoholic potash to absorb carbon disulphide and carbon dioxide, and then the residual gas exploded with oxygen. Only a slight trace of sulphur dioxide could be detected, but there was a considerable quantity of carbon dioxide present. Undoubtedly, therefore, carbon monoxide was present in the gas.

Experiments were also tried without nitrogen: the whole apparatus was swept out by a current of carbon disulphide vapour obtained by boiling the liquid under reduced pressure, and the copper then heated in the stream of carbon disulphide vapour. The resulting gas was again found to contain some carbon dioxide and carbon monoxide.

In some experiments the copper was reduced by alcohol as mentioned by Thomsen, whilst in others it was heated strongly for some time in a current of hydrogen.

Manchester, January 6th, 1904.

## NOTE ON THE CONNECTION BETWEEN NEGATIVE ELECTRICITY AND THE VALENCE OF ATOMS.

By GEOFFREY MARTIN, B.Sc.(Lond.).

IN the CHEMICAL NEWS last April (1903, lxxvii., 162), I suggested that it was the negative electricity which an atom contained that determined the degree of valence with which an element can act.

That the addition of electricity to an element can often cause it to temporarily assume degrees of valence otherwise unattainable under ordinary conditions is well known.

For example, a silent electric discharge through a mixture of NO and O cooled to a low temperature causes the formation of NO<sub>3</sub>—the N atom here acting with a valence of six—a valence which it seems incapable of assuming in any other compound.

SO<sub>2</sub> and O under similar conditions unite to form S<sub>2</sub>O<sub>7</sub>—S here acting as S<sup>VI</sup>, also an otherwise unknown grade of valence.

The precise connection between the electric field in which an element is placed and the degree of valence with which the element acts seems to be indicated by the following facts:—

"When a solution of sulphuretted hydrogen in water is decomposed by an electric current, the sulphur is deposited on the positive pole, and has therefore an electro-negative nature, and this sulphur is soluble in CS<sub>2</sub>. When a solution of sulphurous acid is decomposed in the same manner, the sulphur is deposited on the negative pole, and is therefore electro-positive; and the sulphur so deposited is insoluble in CS<sub>2</sub>. The sulphur which is combined with metals must have the properties of the sulphur contained in sulphuretted hydrogen, whilst the sulphur combined with chlorine is like that which is combined with oxygen in sulphurous anhydride.

"Hence Berthelot recognises the presence of soluble sulphur in metallic sulphides, and of the insoluble modification of amorphous sulphur in sulphur chloride.

"Cloeze showed that the sulphur precipitated from solutions is either soluble or insoluble, according to whether it separates from an alkaline or acid solution" (Mendeleeff, "Principles of Chemistry," 1897, ii., 207).

It appears, therefore, that the divalent S in  $\text{H}_2\text{S}$  (and in metallic sulphides generally) is deposited on the anode, and therefore comes from the cathode. Whereas the polyvalent S in  $\text{H}_2\text{SO}_3$  (and in combinations of S with non-metals generally) is deposited on the cathode, and therefore comes from the anode.

Metals behave in the same way. For example, Hittorf ("Über das Verhalten des Chroms," *Zeit. für Elektrochemie*, 1899, No. 1) shows that when metallic Cr is made the cathode it is in a divalent state, since it dissolves as  $\text{CrX}_2$ ; whereas when anode it is hexavalent, since it dissolves as  $\text{CrO}_3$ . Iron shows a similar behaviour.

It appears, therefore, that an element, whether metallic or non-metallic in nature, develops a high valence when made anode (i.e., to receive negative electricity), and a low valence when made cathode (i.e., when negative electricity is drained away from it).

This strongly supports the view that the valency of an element is connected in the way previously indicated with the quantity of negative electricity it contains.

Kiel, December 7, 1903.

### PRECIPITATION OF SOME ALKALOIDS BY NITRATE OF URANIUM; REACTION OF MORPHINE.

By J. ALOY.

THE use of general reagents, such as chloride of platinum or gold, iodised potassic iodide, double iodides of potassium and mercury, or of potassium and bismuth, phosphotungstic acid, &c., for the detection of alkaloids, offers serious difficulties in many cases.

Occasionally these reagents precipitate foreign bodies, ammoniacal salts, or albumin; also the precipitates formed are often badly adapted for the recovery of the alkaloid.

I was anxious to know whether a certain number of vegetable bases could not be identified by making use of their alkaline function. I decided on using nitrate of uranium,  $(\text{NO}_3)_2\text{UO}_2 \cdot 6\text{H}_2\text{O}$ , as the reagent. The principal reasons I had for making this choice are as follows:—In the presence of free ammonia, uranium gives a precipitate consisting of an insoluble uranate which by simple calcination is transformed into the oxide,  $\text{U}_3\text{O}_8$ . The weight of the oxide enables us to know immediately the amount of the base present in the compound.

Further, nitrate of uranium in dilute solution is without action on the salts and on organic matters. Finally, this salt is very soluble in alcohol, ether, and the reagents in general use for the extraction of the alkaloids.

Most of the alkaloids in etherial or aqueous alcoholic solution act on nitrate of uranium in the same manner as ammonia.

I have examined the following ones, which all give a precipitate of the uranate:—Pyridine, narcotine, papaverine, codeine, thebain, narcein, quinine, cinchonine, cinchonidine, strychnine, brucine, cocaine, pelletierine, aconitine, atropine, and cicutine.

Cafein, theobromine, and asparagine are not precipitated; as for morphine it gives a characteristic coloured reaction.

To effect the precipitation of the alkaloids, it is best to take a solution of the nitrate diluted to 5 per cent, and neutralised very carefully with ammonia until precipitation just begins. The nitrate is then added drop by drop to the solution of the alkaloid so long as the precipitate is formed.

The sensitiveness of this method is fairly great when the alkaloid is present in a small amount of the solvent. We are able to detect a tenth of a m.grm. of most of the alkaloids that I have examined. The precipitate once formed remains easily distinguishable when the solution is diluted.

If the solution under examination is very dilute, a cloudiness is produced which is transformed slowly into a precipitate.

This transformation is facilitated by keeping the temperature up to  $60^\circ$  or  $70^\circ$ .

The uranates of the alkaloids have the general properties of the alkaline uranates. They are insoluble in water and alcohol inversely as most of the salts of the alkaloids are.

Though amorphous at the moment of their formation, several of these precipitates take a crystalline form after a little time. They all have a more or less accentuated yellow colour.

I have analysed the compounds of uranic acid with pyridine, strychnine, brucine, and codeine by estimating the uranium as oxide,  $\text{U}_3\text{O}_8$ , and the amount of alkaloid from the volume of nitrogen measured by Dumas's method. None of these compounds corresponds to the normal acid,  $\text{UO}_3 \cdot \text{H}_2\text{O}$ ; they consist of biuranates with the general formula  $\text{U}_2\text{O}_8\text{H}_2 \cdot 2\text{Alk} + \text{Aq}$ . In every case analysis shows a deficit of nitrogen which is increased by washing.

The uranates enable us to recover the original alkaloids very easily. It suffices to treat them with an alkaline bicarbonate, which sets the alkaloid at liberty and dissolves the uranic acid. The alkaline uranates are also dissolved under the same conditions.

*Reaction of Morphine.*—Morphine and its salts in the presence of dilute nitrate of uranium give a beautiful red colouration. This reaction is due to a reduction of the uranium salt by the phenolic function of this base. It is not produced by any of the other alkaloids I have mentioned. Thus it enables us to identify morphine in a mixture.

The colouration is distinctly red with quantities of morphine greater than 5 m.grms.; it changes to an orange tint when the proportion of alkaloid diminishes.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 12.

### THE USE OF BINOXIDE OF LEAD IN ANALYSIS.

By M. St. BOGDAN.

My bibliographical research, in so far as concerns the reactions which take place between binoxide of lead and sulphuretted hydrogen in aqueous solution, and between binoxide of lead and solutions of different metallic sulphides, has been unsuccessful.

The experiments undertaken with a view to deciding this question will be published later on.

In the meantime, I propose describing the experiments made with the object of effecting the rapid elimination of sulphuretted hydrogen or metallic sulphides from any solution.

I have observed that an aqueous solution of sulphuretted hydrogen is decomposed by powdered binoxide of lead in the cold, with the formation of sulphide of lead. I made use of this reaction to free an alcoholic solution from traces of sulphuretted hydrogen, which was present as an impurity. After having digested the solution for three hours with binoxide of lead in excess, there was no longer any perceptible odour of sulphuretted hydrogen. The reaction is still more rapid with the metallic sulphides, and especially so with sulphide of ammonium; and in these cases it is always accompanied with a considerable evolution of heat. M. Vraciu was kind enough to inform me of an observation made by him, that a mixture of a concentrated solution of sulphide of ammonium and powdered binoxide of lead ignites spontaneously when an excess of the binoxide is thrown suddenly into the solution of the sulphide.

On the above facts briefly related is founded the method I propose for the decomposition of the sulphide of ammonium that is present in excess in the solution, after the precipitation in the form of sulphides of metals, of which the salts are not precipitable by sulphuretted hydrogen in acid solution.

The great disadvantage of the method employed up to



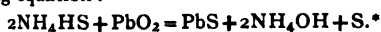
the present for removing the excess of sulphide of ammonium after the precipitation of the said metals—decomposition of the sulphide by hot hydrochloric acid—rests in the slowness of the operation; in fact, it is necessary to heat for a long time to eliminate the sulphuretted hydrogen completely, and the filtration, which is always indispensable, is rarely very rapid on account of the sulphur which is deposited in a very finely divided state. There is also a considerable increase in volume, which necessitates the concentration of the liquid on the water-bath before searching for the bases of the following groups. On the other hand, by adding powdered binocide of lead to the solution containing the sulphide of ammonium, my experiments have shown that this substance transforms even the last traces of the sulphide of ammonium into ammonia with the precipitation of the sulphide of lead. The reaction goes on in the cold, though it is preferable to heat for a few minutes on the water-bath.

Two series of experiments that I am about to summarise show still better the advantages of this method.

In the first series of experiments I took 10 c.c. of a saturated solution of freshly prepared sulphide of ammonium, and after having diluted up to 100 c.c., I heated this solution for five minutes with binocide of lead in excess. After cooling the solution, I filtered it. The resulting liquid was perfectly colourless; it did not give off any odour of sulphide of ammonium, and it was not possible to detect any trace of sulphur by any reaction whatever. On evaporating a small quantity of the liquid to dryness in a platinum crucible, I found that it did not leave any residue; on the other hand, the solution contained ammonia.

The blackish-brown residue is a mixture of sulphide of lead, binocide of lead, and sulphur.

The reaction which takes place may be expressed by the following equation:—



The importance of these experiments rests in the fact that all traces of sulphide of ammonium are destroyed, and there remains ammonia in solution as the product of the reaction.

The second series of experiments was undertaken to prove that the bases of the following groups are not carried down by this precipitation. To 50 c.c. of a normal solution of chloride of barium, I added 10 c.c. of a concentrated solution of sulphide of ammonium, and after precipitation with the binocide of lead while heating, I filtered. The filtrate comprised the washings of the residue, and after acidulation with hydrochloric acid it was precipitated with sulphuric acid.

The quantity of sulphate of barium found showed that the whole of the chloride of barium was present in solution after the precipitation with binocide of lead.

This operation was repeated several times on solutions containing a mixture of chlorides or nitrates of calcium, barium, and strontium; the solution, after similar treatment, always contained the same quantity of salts in solution.

It results from these experiments that on decomposing sulphide of ammonium in this manner, we can get rid of the whole of the sulphur, and that the ammonia, which is even necessary for the subsequent treatment of the substance under analysis, remains in solution.

Further, the alkaline-earth bases are not carried down by the insoluble residue, and the filtrate does not contain a trace of lead.

The simplicity of the operation, as well as the rapidity with which it can be carried out, makes it very useful. It may be also observed that this method is both more economical and more hygienic on account of there being no disengagement of sulphuretted hydrogen.

\* The reaction, which I am still examining, seems to be in reality more complicated. In fact, I have found in the residue a colourless crystalline product, insoluble in ammonia, but soluble in nitric acid. It contains lead.

Being persuaded that this method, both simple and convenient, will be of great service to analytical chemists, I think I am right in not longer deferring its publication.

Before long I shall give the results of other researches now in progress on a method for the volumetric estimation of sulphuretted hydrogen and of sulphides, by the use of binocide of lead.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 12.

## BEHAVIOUR OF PHENOLPHTHALEIN IN THE PRESENCE OF BICARBONATES AND OF ALKALINE CARBONATES.

By M. GIRAUD.

PHENOLPHTHALEIN becomes pink in the presence of alkaline carbonates; I have endeavoured to find out up to what point this colouration persists when titrating these salts with sulphuric acid. Now, I have found that with a solution of  $\text{CO}_3\text{Na}_2$ , titrating, for 10 c.c., 10.7 c.c. of molecular sulphuric acid in the presence of tropeolein, I only obtained 5.35 c.c. in the presence of phenolphthalein. Another experiment made with 53 grms. of chemically pure anhydrous  $\text{CO}_3\text{Na}_2$ , titrated 50 c.c. with molecular acid in the presence of tropeolein, I only obtained 25 c.c. with phenolphthalein.

Through a solution of carbonate of soda red to phthalein, I passed a current of  $\text{CO}_2$ , previously washed, and after a certain time the solution lost its colour; I made the same experiment with bicarbonate of soda containing traces of carbonate, and the decoloration was complete after a few minutes; this shows that phenolphthalein becomes pink in the presence of alkaline carbonates, and loses its colour in the presence of the corresponding bicarbonates.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 12.

## THE BEHAVIOUR OF CERIUM, LANTHANUM, NEODYMIUM, PRASEODYMIUM, THORIUM, AND ZIRCONIUM TOWARD ORGANIC BASES.\*

By BURT LAWS HARTWELL.

(Concluded from p. 16).

*p*-Toluidine.—From observations made in the qualitative way, it appeared not improbable that *p*-toluidine might be employed to separate zirconium and thorium from lanthanum, neodymium, and praseodymium. Accordingly, determinations were made in about 100 c.c. of the weak alcoholic solutions, a moderate heat being applied to hasten the reaction. The precipitates formed were quite voluminous and somewhat gelatinous, especially in the case of zirconium, and well adapted for bringing down the accompanying element. On this account one re-precipitation was carried out in each case. The first precipitates dissolved readily in dilute nitric acid, especially if the solutions in the case of zirconium were not allowed to become too warm. The accompanying element was precipitated by ammonia from the combined filtrates. The ignition of the precipitates was conducted with free access of air, in a porcelain crucible usually, and the blast-lamp used till constant weights were obtained. All of the weights, recorded in grms., are given in the condensed form below. The weights given as "required" were obtained by direct precipitation of the elements, separately, and blasting to constant weight, and represent the average of two closely agreeing results. The "combined" oxides represent the sum of the individual oxides. The results obtained in many cases do not agree well with the standard, but it was thought best to include all which, so far as known, were carried through without accident. Some minor differences

\* Contributions from the John Harrison Laboratory of Chemistry. (From author's thesis for Ph.D. degree). From the *Journal of the American Chemical Society*, xxv., No. 11.

in manipulation, such as varying the strength of alcohol, time of digestion, temperature, and volume of solution, were made in different cases. These variations may have influenced the results in some instances.

#### Zirconium and Lanthanum.

| Zirconium oxide— |          | Lanthanum oxide— |          | Combined oxides— |          |
|------------------|----------|------------------|----------|------------------|----------|
| Found            | Required | Found            | Required | Found            | Required |
| 0.1342           | 0.1341   | 0.0399           | 0.0409   | 0.1741           | 0.1750   |
| 0.1301           | 0.1341   | 0.0993           | 0.1023   | 0.2294           | 0.2364   |
| 0.1314           | 0.1341   | 0.1025           | 0.1023   | 0.2339           | 0.2364   |
| 0.1367           | 0.1341   | 0.0954           | 0.1023   | 0.2321           | 0.2364   |

#### Zirconium and Neodymium.

| Zirconium oxide— |          | Neodymium oxide— |          | Combined oxides— |          |
|------------------|----------|------------------|----------|------------------|----------|
| Found            | Required | Found            | Required | Found            | Required |
| 0.1349           | 0.1341   | 0.0413           | 0.0457   | 0.1762           | 0.1798   |
| 0.1439           | 0.1341   | 0.0973           | 0.1142   | 0.2412           | 0.2487   |
| 0.1372           | 0.1341   | 0.1102           | 0.1142   | 0.2474           | 0.2487   |
| 0.1446           | 0.1341   | 0.0952           | 0.1142   | 0.2398           | 0.2487   |

#### Zirconium and Praseodymium.

| Zirconium oxide— |          | Praseodymium oxide— |          | Combined oxides— |          |
|------------------|----------|---------------------|----------|------------------|----------|
| Found            | Required | Found               | Required | Found            | Required |
| 0.1317           | 0.1341   | 0.0420              | 0.0458   | 0.1737           | 0.1799   |
| 0.1344           | 0.1341   | 0.1099              | 0.1144   | 0.2443           | 0.2485   |
| 0.1312           | 0.1341   | 0.1129              | 0.1144   | 0.2441           | 0.2485   |
| 0.1304           | 0.1341   | 0.1014              | 0.1144   | 0.2318           | 0.2485   |

#### Thorium and Lanthanum.

| Thorium oxide— |          | Lanthanum oxide— |          | Combined oxides— |          |
|----------------|----------|------------------|----------|------------------|----------|
| Found          | Required | Found            | Required | Found            | Required |
| 0.1254         | 0.1247   | 0.0407           | 0.0409   | 0.1661           | 0.1656   |
| 0.1222         | 0.1247   | not det.         | 0.1023   | —                | —        |
| 0.1246         | 0.1247   | 0.1035           | 0.1023   | 0.2281           | 0.2270   |
| 0.1243         | 0.1247   | 0.1034           | 0.1023   | 0.2277           | 0.2270   |

#### Thorium and Neodymium.

| Thorium oxide— |          | Neodymium oxide— |          | Combined oxides— |          |
|----------------|----------|------------------|----------|------------------|----------|
| Found          | Required | Found            | Required | Found            | Required |
| 0.1254         | 0.1247   | 0.0464           | 0.0457   | 0.1718           | 0.1704   |
| 0.1232         | 0.1247   | 0.1146           | 0.1142   | 0.2378           | 0.2389   |
| 0.1242         | 0.1247   | 0.1142           | 0.1142   | 0.2384           | 0.2389   |
| 0.1242         | 0.1247   | 0.1129           | 0.1142   | 0.2371           | 0.2389   |
| 0.1248         | 0.1247   | 0.1118           | 0.1142   | 0.2366           | 0.2389   |

#### Thorium and praseodymium.

| Thorium oxide— |          | Praseodymium oxide— |          | Combined oxides— |          |
|----------------|----------|---------------------|----------|------------------|----------|
| Found          | Required | Found               | Required | Found            | Required |
| 0.1253         | 0.1247   | 0.0471              | 0.0458   | 0.1724           | 0.1705   |
| 0.1214         | 0.1247   | 0.1147              | 0.1144   | 0.2361           | 0.2391   |
| 0.1238         | 0.1247   | 0.1160              | 0.1144   | 0.2398           | 0.2391   |
| 0.1246         | 0.1247   | not det.            | 0.1144   | —                | —        |

It will be noticed that the results with zirconium are not so satisfactory as those with thorium. This cannot be attributed to any difference in manipulation for they were obtained under similar conditions, the usual practice being to carry on the six separations at one time.

A tendency on the part of zirconium to resist separation from other elements is again indicated here. This tendency is exhibited by its precipitation being retarded by the presence of another element, and by the fact that when once brought down it frequently is contaminated by that element. In the attempts to separate zirconium from thorium by *m*-chloraniline, it was repeatedly noticed that conditions of digestion, sufficient to cause precipitation of zirconium by itself, were inadequate when thorium was present, and when the precipitate was thrown down it was badly contaminated with thorium. Similar observations were made when formin was used to effect their separation; in that case all of the thorium came down with the zirconium under conditions which failed to precipitate thorium when alone. It might be supposed that this peculiar behaviour of zirconium would not be noticed upon passing from thorium to the more basic elements, but a number of the results obtained in connection with lanthanum and praseodymium show incomplete precipitations

of zirconium, while neodymium was so inclined to come down with zirconium that a satisfactory separation could only be expected when all possible precautions had been taken to prevent it.

The oxides obtained by ammonia from the filtrates weighed less in the separations from zirconium than in those from thorium. If one attempts to attribute this difference to the greater solvent action of the filtrates from the zirconium, the only respect that suggests itself in which the two sets of filtrates differed is that they probably contained different amounts of the precipitant, *p*-toluidine. More of this reagent was necessary to effect a precipitation in the solutions containing thorium than in those in which zirconium was present, and it is probable that in carrying out the two precipitations, as was done in each instance, considerably more *p*-toluidine accumulated in the filtrates from the thorium. Sufficient attention was not given to this point while the determinations were being made to warrant an opinion as to the extent of its influence and it is merely offered as a possible explanation. As both the first and second precipitations were usually made from about 100 c.c. of solution, the combined filtrates and washings, in which the precipitations by ammonia were made, were of considerable volume, and the question of the solvent effect of all this solution upon the precipitate produced by ammonia presented itself at the time of the determinations. The final filtrates were usually partially distilled, the bulk of the alcohol and ammonia being obtained in the distillate, from which the alcohol was recovered. More ammonia was repeatedly added to the undistilled portions to see if further precipitation would take place. In a few instances the entire solution was evaporated to dryness and the residue ignited and weighed. The weights obtained substantially made good the discrepancies between the combined oxides "obtained" and "required," but as no blank determinations were made it was not proved that some of this may not have come from extraneous sources.

As previously stated, the recorded results were obtained by the use of the blast-lamp till practically constant weights resulted. The writer is of opinion, however, that errors may have occurred in this connection due to a constant composition not always being obtained, especially with lanthanum, neodymium, and praseodymium. The weights obtained, by blasting the first two, were frequently 10 per cent lower than those obtained by igniting over a Bunsen burner; there was less difference with praseodymium. Hitchcock found that neodymium and praseodymium sesquioxides by gentle ignition with free air access, gradually took on oxygen till the weights approximated those represented by the formula  $M_2O_7$  (*Journ. Am. Chem. Soc.*, xvii., 525).

Herzfeld (*"Chemie der seltenen Erden,"* p. 63) speaks of neodymium oxide as having a blue colour and assigns to it the composition  $Nd_2O_3$ , and again (*Ibid.*, p. 122), in an abstract from Shapleigh's investigation, mentions a suboxide as a grey powder and a peroxide as a dirty grey powder. The oxide obtained in the present investigation was invariably of a light brown colour resembling cocoa powder, after ignition with the Bunsen burner, and was usually changed to a light blue or light purple colour by blasting. It was often necessary to pulverise and blast a long time before the bluish colour was uniformly obtained; indeed, in some instances part of the oxide still retained the cocoa colour, even though by excessive blasting practically no change in weight resulted. In view of the inadequate and somewhat conflicting information concerning neodymium oxides, the composition of the oxide obtained in any particular case must receive careful attention. The change of colour produced by blasting the lanthanum and praseodymium oxides was not sufficient to indicate a change in composition, but the weights, especially with lanthanum, were considerably reduced and the certainty of a definite composition should also be established in this instance.

The most that was attempted, during the present work, with any of the oxides, was to compare the weights obtained in the course of the analyses with those obtained

by direct precipitation from the aqueous solutions by ammonia, and blasting until they were constant within a few tenths of a milligram. It would seem as though the oxides obtained in this manner would be of constant composition, but it is not improbable that small variations may have occurred, and that lack of nice conformity may occasionally be attributed in part to this cause. The mechanical condition of the oxides previous to blasting was not always the same, and it seems not improbable that this fact may be of some importance where intense blasting is resorted to in order to remove the last traces of oxygen necessary to effect a complete transition from an oxide which, for example, is stable at the temperature reached by the Bunsen burner to one of a lower state of oxidation which may be the stable form at the temperature produced by the more intense heat of the blast-lamp. Judging from the experience of Hitchcock, and from the fact that blasting invariably reduced the weights, it seems probable that, in the case of some of the elements, at least, an oxide of a higher state of oxidation was more stable over the Bunsen burner than over the blast-lamp. Such behaviours have been noticed with some of the more common elements.

### THE DETERMINATION OF BENZENE IN ILLUMINATING GAS.\*

By L. M. DENNIS and J. G. O'NEILL.

(Concluded from p. 15).

In the series of experiments described in the following pages, the analyses were carried out in a "simple" Hempel burette provided with a water jacket, and water was used as the confining liquid.

Experiments were first made to ascertain whether benzene vapour mixed with air can be quantitatively absorbed by the ammoniacal nickel solution just described. The gas mixture was first passed into a pipette containing the ammoniacal nickel solution and was shaken with that reagent for three minutes. It was then drawn back into the burette, passed into a pipette containing mercury and 5 c.c. of 5 per cent sulphuric acid, and was shaken for three minutes, being then drawn back and measured.

TABLE III.

|                                                                    | I.<br>C.c. | II.<br>C.c. | III.<br>C.c. |
|--------------------------------------------------------------------|------------|-------------|--------------|
| Air taken . . . . .                                                | 52.7       | 48.6        | 51.3         |
| Air plus benzene . . . . .                                         | 56.4       | 52.4        | 55.2         |
| After shaking with ammoniacal<br>nickel nitrate solution . . . . . | 53.2       | 49.0        | 51.9         |
| After shaking with 5 per cent<br>sulphuric acid . . . . .          | 52.7       | 48.6        | 51.3         |
| Benzene taken . . . . .                                            | 3.7        | 3.8         | 3.9          |
| Benzene found . . . . .                                            | 3.7        | 3.8         | 3.9          |

The above results show that benzene vapour is quantitatively absorbed by treatment with an ammoniacal nickel solution prepared as above, when the gas mixture is shaken with the reagent for three minutes. Results of approximate accuracy can be obtained by using a simple burette without water jacket, but there seems to be a slight heating of the gas mixture resulting from the absorption of the ammonia gas by the dilute sulphuric acid, this rise in temperature being sufficient to cause appreciable error in the final reading. To ascertain whether the time necessary for the absorption of the benzene could be reduced, other mixtures of benzene and air were prepared and were shaken with the absorbent for one minute, for two minutes, and for three minutes. The results, which need not here be inserted, showed that a three-minute shaking of the gas mixture with the absorbent is necessary for the complete removal of the benzene.

The next point to be ascertained was whether the nickel solution would absorb other constituents of illuminating gas, and would so interfere with their determination as to render its use for the absorption of benzene impossible.

\* From the *Journal of the American Chemical Society*, xxv., No. 5.

Measured amounts of carbon dioxide were mixed with measured amounts of air, and this mixture was passed into a pipette containing the nickel solution. As was to be expected, the carbon dioxide was completely removed. It had already been found that the nickel solution had no effect upon air, and consequently the absorption of oxygen in the illuminating gas by this reagent did not need further examination. To ascertain whether carbon monoxide and the unabsorbable residue, consisting chiefly of methane, hydrogen, and nitrogen, is affected by the nickel solution, the heavy hydrocarbons were removed from 100 c.c. of illuminating gas by means of fuming sulphuric acid and potassium hydroxide, and the residue was then shaken for three minutes with the nickel solution and then with the dilute sulphuric acid. No diminution in volume resulted. From these experiments it appears that the ammoniacal nickel solution beyond removing, wholly or in part, the hydrocarbons other than methane, absorbs no other gas except carbon dioxide, but the fact that this last-named gas is removed by the reagent makes it impossible to use the nickel solution for the removal of benzene and allied carbons, and still adhere to the order that is usually followed (Hempel-Dennis's "Gas Analysis," 1902, p. 282). In the customary procedure the hydrocarbon vapours are first absorbed by alcohol, then carbon dioxide by caustic potash, and then the so-called heavy hydrocarbons by fuming sulphuric acid. Hempel and Dennis state that carbon dioxide may not first be removed by means of potassium hydroxide because benzene is soluble in that reagent (*Ibid.*, p. 281). Experiments were made to ascertain whether this statement is correct. A measured volume of air was mixed with a measured volume of benzene vapour, and this mixture was passed over into a pipette containing potassium hydroxide. No diminution in volume resulted. It therefore appears that the absorption of carbon dioxide by potassium hydroxide may properly precede the absorption of benzene. To be perfectly sure upon this point, however, the following experiment was made:—Three Muencke wash-bottles containing clear barium hydroxide solution were connected together and illuminating gas was passed through this chain. The carbon dioxide present in the gas was completely removed by the reagent in the first bottle. The gas issuing from the third bottle was therefore free from carbon dioxide, but could safely be assumed to contain some of all of the other constituents of illuminating gas. 100 c.c. of the gas issuing from the third bottle was drawn off into a Hempel burette and was then passed over into the caustic potash pipette. No decrease in volume took place. A repetition of this experiment gave the same result, and there was thus obtained confirmation of the statement made above, that potassium hydroxide removes nothing from illuminating gas except carbon dioxide.

Experiments were next undertaken to ascertain, if possible, the nature of the hydrocarbons removable by alcohol and by the ammoniacal nickel solution. These hydrocarbons probably consist largely of ethylene, propylene, and benzene. Ethylene was prepared by treating pure ethylene bromide with a zinc-copper couple. The purity of the gas was tested by passing it into fuming sulphuric acid, the confining water in the burette having first been saturated with the ethylene. Ten c.c. of the gas left a residue of 0.2 c.c. showing a purity of 98 per cent. Measured volumes of air were now mixed with measured volumes of this gas, and the mixture was then passed into a pipette containing the nickel solution and then into a pipette containing mercury and 5 c.c. of 5 per cent sulphuric acid. The results obtained were:—

TABLE IV.

|                                                                    | I.<br>C.c. | II.<br>C.c. | III.<br>C.c. |
|--------------------------------------------------------------------|------------|-------------|--------------|
| Air taken . . . . .                                                | 49.2       | 51.5        | 48.0         |
| Air plus ethylene . . . . .                                        | 58.0       | 60.8        | 54.2         |
| After shaking with ammoniacal<br>nickel nitrate solution . . . . . | 58.8       | 62.4        | 55.1         |
| After shaking with 5 per cent<br>sulphuric acid . . . . .          | 57.9       | 60.9        | 54.2         |

It therefore appearing that ethylene is not taken up by the nickel solution, experiments were next carried out to ascertain whether benzene and ethylene could be separated by means of the nickel solution. The results of these experiments follow:—

TABLE V.

|                                                                                               | I.<br>C.c. | II.<br>C.c. | III.<br>C.c. | IV.<br>C.c. |
|-----------------------------------------------------------------------------------------------|------------|-------------|--------------|-------------|
| Air taken .. .. .                                                                             | 50.2       | 60.7        | 48.9         | 54.4        |
| Air plus benzene .. .. .                                                                      | 53.0       | 64.2        | 51.1         | 57.2        |
| Air plus benzene plus ethylene ..                                                             | 57.6       | 68.7        | 57.2         | 64.4        |
| After shaking with ammoniacal nickel nitrate solution and then with 5 per cent sulphuric acid | 54.7       | 65.2        | 54.9         | 61.6        |
| Benzene taken .. .. .                                                                         | 2.8        | 3.5         | 2.2          | 2.8         |
| Benzene found .. .. .                                                                         | 2.9        | 3.5         | 2.3          | 2.8         |
| After shaking the residue with absolute alcohol and then with water .. .. .                   | 53.4       | 64.6        | 54.7         | 61.1        |
| After treatment with fuming sulphuric acid and then with potassium hydroxide .. .. .          | 50.7       | 61.3        | 49.7         | 55.3        |
| Ethylene taken (88 per cent pure)                                                             | 4.6        | 4.5         | 6.1          | 7.2         |
| Ethylene found by alcohol .. ..                                                               | 1.3        | 0.6         | 0.2          | 0.5         |
| Ethylene found by fuming sulphuric acid and potassium hydroxide after alcohol .. ..           | 2.7        | 3.3         | 5.0          | 5.8         |
| Total ethylene found .. .. .                                                                  | 4.0        | 3.9         | 5.2          | 6.3         |
| Ethylene taken (corrected volume) .. .. .                                                     | 4.04       | 3.9         | 5.36         | 6.3         |

The results in Table V. show that a satisfactory separation of benzene from ethylene and probably from the other hydrocarbons of the ethylene series may be effected by first shaking the gas mixture with the ammoniacal nickel nitrate solution for three minutes, and then with a 5 per cent solution of sulphuric acid for the same length of time.

It was hoped that it might be possible to remove the ethylene series of hydrocarbons by means of absolute alcohol before subjecting the illuminating gas to treatment with fuming sulphuric acid. If this were possible, the hydrocarbons would be divided, analytically, into three distinct groups, and a much clearer idea of the illuminants in the gas could then be obtained from the results of a volumetric analysis than is possible under the present methods. The results in Table V. have shown that the absorption of ethylene by alcohol is far from complete, and, moreover, that the results of such absorption are by no means constant. It was thought desirable, however, before abandoning the separation by alcohol to try the method with illuminating gas, and in Table VI. the results of these experiments are given.

TABLE VI.

|                                                                                   | I.<br>C.c. | II.<br>C.c. | III.<br>C.c. | IV.<br>C.c. |
|-----------------------------------------------------------------------------------|------------|-------------|--------------|-------------|
| Carbon dioxide .. .. .                                                            | 1.2        | 1.2         | 1.1          | 1.1         |
| Benzene by nickel solution and dilute sulphuric acid .. ..                        | 1.2        | 1.0         | 1.0          | 1.0         |
| Hydrocarbons removed by absolute alcohol and water .. ..                          | 1.5        | 0.3         | 1.3          | 0.5         |
| Heavy hydrocarbons removed by fuming sulphuric acid and potassium hydroxide .. .. | 2.3        | 3.5         | 2.6          | 3.4         |
| Total hydrocarbons absorbed by the three reagents ..                              | 5.0        | 4.8         | 4.9          | 4.9         |

These results demonstrate conclusively that absorption by alcohol can not be employed for the purpose in hand, and that at the present time the gas analyst must content himself with the absorption of benzene by the nickel solution and the subsequent absorption of the so-called heavy hydrocarbons by fuming sulphuric acid. In Table VII. are given four analyses of illuminating gas, showing the accuracy of the determinations of carbon dioxide, benzene, and the heavy hydrocarbons.

TABLE VII.

|                                                                                     | I.<br>C.c. | II.<br>C.c. | III.<br>C.c. | IV.<br>C.c. |
|-------------------------------------------------------------------------------------|------------|-------------|--------------|-------------|
| Carbon dioxide .. .. .                                                              | 1.2        | 1.2         | 1.2          | 1.2         |
| Benzene by nickel solution and dilute sulphuric acid .. ..                          | 1.0        | 1.0         | 0.9          | 0.9         |
| Heavy hydrocarbons removed by fuming sulphuric acid and potassium hydroxide .. .. . | 4.0        | 3.9         | 3.9          | 4.0         |
| Total hydrocarbons .. ..                                                            | 5.0        | 4.9         | 4.8          | 4.9         |

Before the ammoniacal solution of nickel nitrate and the 5 per cent solution of sulphuric acid are used for the absorption of benzene, the reagents should, of course, be saturated with the other constituents of the gas mixture in the usual manner (Hempel-Dennis's "Methods of Gas Analysis," 1902, p. 119). If the reagents in the pipettes have been used for analysis of illuminating gas, they should not be used in the examination of generator gas or of any other gas mixture differing appreciably from the illuminating gas, for the gases that have been dissolved by the reagent when it was shaken with the illuminating gas would escape into another superimposed gas mixture, if this latter did not contain these dissolved gases at approximately the same partial pressure as that at which they existed in the gas with which the reagent was first shaken. Consequently the gas pipettes should be filled with fresh solutions of ammoniacal nickel nitrate and sulphuric acid whenever the gas mixture to be analysed differs markedly from that for which the reagents had previously been used.

The results may briefly be summarised as follows:—

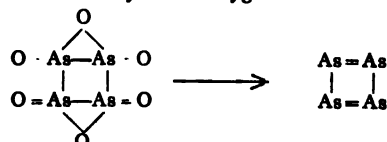
1. Under the described conditions, alcohol does not completely remove either benzene or ethylene from gas mixtures.

2. The use of an ammoniacal solution of nickel nitrate furnishes a rapid and exact method for the determination of benzene in mixtures of that substance with air and ethylene, and in coal gas.

The authors would recommend that in the analysis of illuminating gas (coal gas), the order of procedure be:—(a) The absorption of carbon dioxide by potassium hydroxide; (b) the absorption of benzene by the ammoniacal solution of nickel nitrate above described; (c) the absorption of the "heavy hydrocarbons" by fuming sulphuric acid; (d) the absorption of oxygen by alkaline pyrogallol or by phosphorus; (e) the absorption of carbon monoxide by cuprous chloride; and (f) the determination of the methane and hydrogen.

The authors have been unable to try this new method on any commercial gas mixture other than the local supply of illuminating gas. They would therefore earnestly request chemists using the method on other gas mixtures to communicate to them the results of such analyses and call their attention to any difficulties that may arise.

**The Constitution of Sesquioxide of Arsenic.**—H Erdmann.—By reducing vitreous arsenious acid with zinc powder in the presence of sulphide of carbon and of saline solutions, such as solutions of borax, chloride of ammonium, and chloride of magnesium, we obtain a small quantity of yellow arsenic (see below). The author, having shown that this yellow arsenic corresponds to a molecular weight of  $As_4$ , arrives at the formula  $As_4O_6$  for arsenious acid. In this body the atom of arsenic appears to be pentavalent; it becomes trivalent by loss of oxygen.



Vitreous arsenious acid.

Yellow arsenic.

—*Zeit. Anorg. Chem.*, vol. xxxii., p. 453.

THE THERMO-CHEMISTRY OF THE THEORY OF ELECTROLYTIC DISSOCIATION.\*

By JOSEPH W. RICHARDS.

*Heat of Ionisation.*

THE theory of electrolytic dissociation assumes that in dilute solutions of electrolytes the salts are already potentially split up into their positive and negative ions. The first time that I read this statement of the theory, my first thought was:—"Where does the energy come from to cause this dissociation?" This query has been firmly lodged in my mind ever since, and has not been satisfactorily answered by the numerous treatises on electrochemical theory which have since appeared.

The heat of solution in excess of water is usually not a large quantity. The molecular heat of formation of solid potassium chloride is, for instance, 105,700 calories, while if this is dissolved in excess of water 4500 calories are absorbed. If, then, we assume that the solid salt is composed of undissociated molecules, and the dilute solution of ionised molecules only, then the heat absorbed in the process of ionising the solid molecules cannot be over 4500 calories, and therefore bears no quantitative relation at all to the heat of formation of the solid molecules. In fact, we have been overlooking the physical change of state, for the solid salt certainly absorbs heat in changing to the liquid or gaseous molecule in which it occurs in solution, and this change is responsible for part of the 4500 calories absorbed in the act of solution. The heat of ionisation of undissociated molecules of KCl in solution into completely ionised molecules can therefore be but a part of 4500 calories, if it has any real value at all, and the ultimate conclusion is evident that, whatever the energy required to ionise molecules of salt in solution may be, it is not of the same order of magnitude, nor can it be in any way identified with the heat of formation of the salt from its constituent elements. It follows that the act of ionisation, whatever it may be, is certainly not a destruction or dissociation of the compound *qua* compound, but that from the energy standpoint potassium chloride is as really a compound of potassium and chlorine when ionised in dilute solution as when in the solid condition.

There has been a great deal of uncertainty about this question, and the leading exponents of the dissociation theory leave the subject in a very hazy condition. To remedy this, we must stop thinking that a molecule ceases to be a compound when it is dissolved in dilute solution, or ionised. Whatever the ions are, they are not the chemical constituents from which the compound was formed, and the separation of the molecule into ions is not a destruction of the compound or a resolution of it into its chemical constituents. Ostwald says in his "Grundlinien der Anorganischen Chemie," 1900, p. 210:—"Auch in festen salzen, die nicht elektrolytisch dissociirt sind, befinden sich die Bestand-theile dem Ionenzustande viel näher, als dem der freien Elemente. Dies geht daraus hervor, dass der Uebergang der festen Salze in den Ionenzustand bei der Auflösung in Wasser im allgemeinen nur unbedeutende Wärmewirkungen (meist sogar Wärmefreisetzen) ergibt." In other words, Ostwald recognises that since solid salts, which are not electrolytically dissociated, dissolve in water with very slight thermal effects, that the solid molecule of an undissociated compound is nearer to the ionised condition than to the constituent elements from which it was formed. If we turn this statement around, it is that the condition of complete ionisation is much nearer to the undissociated solid molecule than it is to the free constituent elements of that molecule.

What then is the ionised condition? We can say with certainty, for one thing, that it is a condition of the compound, and not a condition in which the compound has ceased to exist and is resolved into its constituent elements.

What is then the heat of ionisation? It is the heat absorbed in a change of state of the compound, and is, as far as we can gather, more a physical change of state than a chemical one. It must not be confounded with the heat of solution, for that includes the change of state from the solid molecule to the dissolved ions: neither can it be completely identified with the heat of dilution, for while during dilution molecules in solution become ionised, yet there are possibly other operations included, such as the making or decomposing of hydrates in solution, and the heat of solution is thus seen to be also a complex quantity.

Arrhenius has calculated heats of ionisation theoretically from thermodynamic considerations, from the change in value of the dissociation constant with temperature. He makes them to be negative quantities even in case where the heat of dilution is positive. It therefore would follow, if these calculations are to be relied upon, that the heat of dilution consists of an absorption of heat due to ionisation plus an evolution of heat due to combination with water. In any case, we are no nearer the solution of the riddle than before.

There is one thermochemical law which gives us pretty firm ground to build upon, viz., the observation that the heats of formation of salts in dilute solution are plainly additive in their nature. This means that the heat of formation of potassium chloride, for instance, is composed of the sum of one value characteristic of potassium and one characteristic of chlorine. All potassium salts therefore differ from all sodium salts, in their heat of formation to dilute solution, by a constant difference—the difference between the constants of potassium and of sodium; while all chlorine salts differ from all bromide salts by a constant difference—the difference between the constants of chlorine and of bromine. This law is in general true, and places the thermochemical constants of the heat of formation of salts to dilute solutions in exactly the same category as the equivalent electrical conductivities of dilute solutions, their specific gravity, specific optical refractive power, and some other physical properties. The truly additive nature of the heats of formation of salts in dilute solutions is a law of the deepest meaning for thermochemistry.

If we consider a dilute solution as containing only ionised molecules, then the heat of formation may be considered, in light of the above thermochemical law, as simply the sum of the heats evolved in the passage of each constituent of the salt from its ordinary molecular condition into the state of being a dissolved ion. The thermochemical constant of a metal would then be regarded as the heat evolved in its passage from the ordinary molecular free state into the state of combination as a dissolved ion. This quantity has been called by Ostwald the *ionisation heat* of the elements, which, it will be observed, is using the term with an entirely different meaning than was given it, as we have been so far considering it. The ionisation heat of a compound is the heat necessary to resolve its dissolved un-ionised molecule into its ions—which operation is not a resolution of the compound into its constituents, and has therefore nothing to do with the heat of formation of the compound. The ionisation heat of an element is the heat given out in changing it from a free, molecular element into the state of a combined dissolved ion. The act of combination is involved in the latter act of ionisation, and the term ionisation here no longer refers to the electrolytic dissociation of the compound.

Considering the term "ionisation heat of the elements," in the sense above given to it, and further considering it as equivalent in value to the thermochemical constants of the elements, we are led to some interesting conclusions. It would follow from this that elements exist in two conditions, viz., the uncombined state and the combined state. Passage into the combined state is accomplished by a change in the intrinsic energy which is characteristic once for all of the element, for a given valency, and independent of any reciprocal action towards the other element or elements with which it is combined; and this combined state is only perfectly attained in dilute solution. Ions in solution are

\* A Paper read at the Fourth General Meeting of the American Electrochemical Society, at Niagara Falls, September 19, 1903.

in this view atoms of the element which have passed completely into the "combined" condition, and in doing so have given out their contribution towards the heat of formation of the compound.

The heats of formation of salts to dilute solutions always give us the sum of the separate heats of combination of its separate ions and their "heats of ionisation." (K, Cl, aq.) = 101,200 calories expresses the fact that the sum of the heats evolved by 39 grms. of solid potassium becoming ionised and 35.5 grms. of chlorine gas becoming ionised, is that many calories. (K<sub>2</sub>, S, O<sub>2</sub>, aq.) = 274,600 calories expresses the sum of the heats of ionisation of 2K-solid and of SO<sub>3</sub> gas (assuming those to be the ions), plus the heat of formation of SO<sub>3</sub> gas. The only difference between the thermochemical constants and the heats of ionisation would be in the case of complex ions such as SO<sub>3</sub>, for instance, in which case the thermochemical constant for SO<sub>3</sub> would be its heat of ionisation (*i.e.* passage from the state of SO<sub>3</sub> gas to SO<sub>3</sub> ion in dilute solution) plus the heat of formation of SO<sub>3</sub> gas from its elements.

In a few cases, such as iodine and mercury, we know approximately the amount of heat necessary to convert the ordinary free element into atoms. I<sub>2</sub> gas = 2I gas absorbs 28,500 calories, but takes place only at a red heat. Hg (liquid) - Hg (monatomic gas) absorbs 13,560 calories, taking place at 355°. We may therefore go slightly further than Arrhenius in these cases, and say that if Hg (liquid) - Hg ion = 20,500 calories, that therefore Hg (monatomic gas) = Hg ion plus 20,500 + 13,560 = + 34,060. This calculation is not exact, because the latent heat of vaporisation of mercury at 18° is not exactly the same as at 355°, being probably somewhat greater. This quantity would be the real heat of ionisation of the element, that is, the heat evolved in conversion of one free gaseous atom into one combined dissolved ion. If this could be determined for a number of elements, it is very likely that some generalisations might become evident. This is the only basis on which the true ionisation heats of elements can be determined, and to deduce them from present data we must know more of the latent heats of fusion and of volatilisation of the elements.

(To be continued),

## A PROPOSED METHOD OF TESTING WOOD TREATED TO RESIST FIRE.\*

By CHAS. F. MCKENNA.

IN the spring of 1902 an effort was made by the Bureau of Buildings of the Borough of Manhattan, New York City, to secure more certitude as to the qualities of the so-called fireproof wood which was being delivered in the city for use in high buildings.

Methods of test which have been in vogue for some time in the Bureau of Buildings were stated to be inadequate for the proper discrimination between well-treated wood and that which was treated only imperfectly and was presumably at times not fireproof in any sense. These methods included the test, long in use in the Navy Department, of exposing shavings to the influence of heat while they rested upon a wire screen, timing the rate of combustion, as well as estimating its extent; also the splinter test as practised in the simple expedient of holding a splinter of wood in and across the flame of a Bunsen burner or upon a red-hot iron plate; also, what may be called the cob-work test, by which prisms of wood about six inches long and one inch, more or less, square, are piled with air-spaces between them and exposed to the flame of a Bunsen burner. There was finally the more meritorious test of Professor Woolsen, of Columbia University, which he calls the timber test, in which specimens of wood one inch square and one foot long are laid in pairs across the top of

a 6-inch gas furnace and exposed to a presumably constant temperature of 1700° F. The depth to which the specimens were charred was subsequently measured and the unburned area used as a basis of comparison.

The fundamental fault with all of these methods of test lies in their neglect of the chemical factors involved in the decomposition of wood under the influence of heat. Nor were proper attempts made to standardise the conditions governing the application of the flame, the access of the air, and the surrounding temperatures, &c. Again, no instrumental means of registering the differences in the results obtained were suggested. In the timber test, the accurate measure of the unburned area does not answer this need, and fails of its purpose where the conditions during the test have so varied.

In the course of the discussion upon this subject which took place about the time mentioned in New York City, there was found to be a demand for an "inspector's test"; that is, for an instrumental and simple test giving results measurable and free from guess and which could be carried on at the place of delivery of the timber. The writer approached this subject from the chemist's side, and his first effort was to undertake distillation tests in closed retorts.

Inasmuch as it was early admitted that these fire-proofing processes could not prevent the destruction of wood by fire operating under its worst aspects, but could only stay the disintegrating effects long enough to present the hope of gaining some advantage over the demon, it seems strange that the search for this test did not then take the form of a study of the reactions proceeding during the inflammation and combustion of the wood, as well as of those accompanying its destructive distillation for the purpose of finding facts leading to more certain means of measuring the retarding values of the various treatments to which wood is subjected for this purpose. The writer outlined such a study by means of destructive distillation of samples of natural and of treated woods; for, although combustion and inflammation with free access of air are undoubtedly the most common conditions of a conflagration, yet the breaking down of the cellulose molecule, of the fundamental tissues of the wood, as well as of its allied organic and mineral constituents, takes place from the centre of large masses as a distillation without air, just as it does also when timber and planking burn at the back of a hot brick wall, or within a metal-cased door or window frame, or where electric wiring passes over wood encased in concrete flooring or in walls. The prosecution of such studies has been stayed through lack of means, but the initial efforts nevertheless have resulted in this proposed method of testing which, with the apparatus offered, seems to be the means for easily making an extended series of critical observations.

Some early distillations made upon charges of wood shavings in 6-ounce glass retorts heated by a triple Bunsen burner developed the fact that when the temperature reaches 250° C. the volatile products cease to be driven off, and temporarily a vacuum is produced if the heat is not promptly raised. This took place in every case in not over three minutes of such heat conditions, and also, under the same conditions, the yield of gas was quite uniform for different woods. To secure higher temperatures in a brief time, the electric wire method was adopted. This led to the invention of an electric test retort, and this in turn, when tried on numerous lots of wood, quickly developed the fact that the little instrument was exactly adapted to meet all the requirements of an inspector's test when the original untreated wood is at hand to compare with the treated, and perhaps also where original samples cannot be obtained. This apparatus is described in my paper presented at this meeting under the title "Electric Test Retort."

In using it for the study of fireproof-wood, it has been my aim to simplify it, as well as the details of the testing, in order that numerous samples of wood could be examined easily and quickly.

\* From the *Journal of the American Chemical Society*, xxv., No. 4.

A large gas burette was provided, having with it a large reservoir for levelling, and this in connection with the electric test retort were all that I found necessary for making critical tests of treated woods. Absorption tubes for the pyroigneous products could be provided, but in the work in hand the necessity was obviated as much as possible by the high initial heat and by providing that the water in the gas burette shall absorb the pyroigneous products until nearly saturated with them.

Violette, as quoted by Sadtler ("Handbook of Industrial Organic Chemistry," second edition, p. 334) in studies devoted to distillation of wood, found that when it is *slowly heated* the water and pyroigneous acids, alcohols, and creosote are all driven off up to and at 280° C. to the extent of 63·8 per cent of the original weight; a large volume of gas up to and at 350° C.; and up to and at 430° C. a total loss of weight of 81 per cent is experienced. But *quickly heated* wood placed at once in the retort at 432° C. lost 91 per cent. Senff (*Ber. d. Chem. Ges.*, xviii., 60) in distilling a variety of woods in 100 kg. charges, found that the uncondensed gases in slowly heated samples varied from 17·17 per cent to 29·23 per cent with an average of 22·64 per cent for fifteen examples of different woods; the uncondensed gases resulting from quickly heated samples varied from 24·07 per cent to 35·56 per cent, with an average for fifteen samples of 31·50 per cent. He found that the charcoal varied from 23·23 per cent to 34·68 per cent, with an average of 27·59 per cent in slowly heated samples, and from 20·20 per cent to 31·59 per cent, with an average of 23·09 per cent, in quickly heated samples. He also found the very important fact that the more common woods, as oak, beech, birch, poplar, and pine, show by the results of the destructive distillation that they resemble one another very much in fundamental composition and give like results in the yield of the respective products.

Hence, we should have in the electric test retort an opportunity to heat quickly to the highest temperature, thus obtaining the maximum gas yield. Fortunately, also, in this test retort, it is not strictly a distillation without access of air; it is really, in a small way, quite a perfect duplication of conditions existing at the beginning of a fire in a closed space, and we have also in this apparatus an opportunity to introduce air in measured quantities, if required.

In conducting the test, a small charge of wood, cut as a little cylinder about 0·5 inch long and 0·25 inch wide, weighing about 500 mg., is placed within the platinum wire basket, the dome is placed upon the apparatus and clamped tightly by suitable means, and the tube leading off from the dome is connected to the gas measuring burette having the usual reservoir tube attached. A current at a difference of potential of 120 volts and a strength of from 7 to 12 amperes is used. Great precautions are needed to keep the current strength steady, and a 7·5 ampere current is the best strength for this experiment. Tests made with the Le Chatelier pyrometer show that the temperature within the coil with a 7·5 ampere current is 680° C.; with a 10 ampere current the temperature rises at the end of three minutes to 840° C. The charge of wood is subjected to the effects of a current of 7·5 amperes for exactly two minutes, the gas being conducted to the burette. Observations of the glow, if any, and of the amount of smoke and the character of same, can all be made and recorded. At the close of the two-minute interval, the current is shut off, the gas burette stop-cock closed, and air admitted to the retort by opening the tubulure at the side. The gas in the burette is cooled, and the volume measured. Or, if a series of tests are being made, the conditions of which are known and invariable, the volume could be read without correction, where so noted. So also, the correction for the volume of air increased by the heat of the contents of the retort can be neglected, for it will be found to be a very constant figure under uniform conditions. Thus, with some experiments in blanks, in heating without the charge with a 7 ampere current for two minutes, the results were 23·6, 22·8, 22·8, 22·6, 22·6 c.c. With the 6·5 ampere

current for two minutes, the results were 21·6, 21·6, and 21·6 c.c. Other experiments gave, with a 7 ampere current for two minutes, 21 c.c. average; with a 7·5 ampere current for two minutes, 24 c.c. average; with an 8 ampere current for two minutes, 26·5 c.c. average; and others with an 8 ampere current for two minutes, 26 c.c. average.

(To be continued).

## CORRESPONDENCE.

### RADIUM.

To the Editor of the Chemical News.

SIR,—It may be worth while to place on record some observations I have made recently in experimenting with radium. I first of all attempted to produce the "scintillations" on a new blende screen by means of radium contained in a sealed glass tube, not knowing at the time that they cannot be caused in this way; but after waiting in the dark for some minutes, I noticed a kind of scintillation resembling occasional meteors on a dark sky, and I have always observed the same appearance (without the tube of radium salt), although the screen had never been brought near radium without intervening glass, and although in some cases the screen had been left for several weeks untouched. I would suggest that these slight scintillations are caused by a spontaneous change in the structure of the blende crystals, which is always taking place, and that the change is brought about suddenly by the impact of the minute particles in the  $\alpha$ -rays.

If radium-barium bromide (I have used 0·1 per cent) is sprinkled on a blende screen, the effect, seen through a magnifying glass, is an appearance like a mass of luminous insects hard at work and quickly moving amongst each other. On shaking off the salt and gently tapping the screen to remove as much as possible, the appearance changes to that of a sky thickly studded with brightly twinkling stars.—I am, &c.,

E. P. PERMAN.

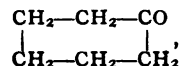
University College, Cardiff,  
January 8th, 1904.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxvii., No. 24, December 14, 1903.

Direct Preparation of Cyclohexanol and Cyclohexanone from Phenol.—Paul Sabatier and J. B. Senderens.—The authors prepare cyclohexanol and cyclohexanone,—



directly from phenol. Reduced nickel is maintained at a temperature of 215–230°, and a mixture of vapour of phenol and excess of hydrogen passed over it. The hydrogenation of the radicle rapidly takes place, tending to produce cyclohexanol. A portion of this is dissociated by the action of the nickel giving the corresponding ketone. The two substances are separated by a delicate operation.

Action of a Mixture of Oxygen and Hydrochloric Acid on Certain Metals.—Camille Matignon.—In a preceding research the author showed that a mixture of oxygen and hydrochloric acid attacked gold, platinum, and tellurium at temperatures far inferior to the temperature of the oxidation of hydrochloric acid gas by oxygen. In the present research he generalises this reaction and finds that



palladium is attacked in the cold by this mixture. In the case of ruthenium, if this metal is placed with the mixture in a sealed tube at 125°, chlorination is complete in a few hours. Iridium at 150° is also attacked in eight to ten hours.

**Constitution and Properties of Silicon Steels.**—Léon Guillet.—The author investigates the constitution and mechanical properties of silicon steels, and shows that—(1) only steels containing less than 5 per cent of silicon can be utilised; (2) silicon steels offer a great resistance to shock after they have been tempered, this resistance is greater for steels rich in carbon; (3) certain anomalies exist between the constitution already established and that proved by the present research, in the case of industrial ferrosilicons and silicon steels, and notably the compound  $\text{Fe}_2\text{Si}$ ; (4) the author's researches, and also those of M. Osmond, seem to prove the existence of two solutions of silicon in iron, one probably having the constitution  $\text{Fe}-\text{Si}$ , the other  $\text{Fe}-\text{Fe}_2\text{Si}$ .

**New Method of Determining the Critical Points of Irons and Steels.**—O. Boudouard.—The author investigates M. Saladin's method of determining the critical points of steel and iron, and his method of utilising the calorific phenomena which accompany the molecular transformations of the metals.

**Meteoric Iron.**—F. Osmond and G. Cartaud.—The authors apply to meteoric iron their method of investigation utilised for the micrographical analysis of irons and terrestrial steels.

**Preparation of Iridium Sesquiselenide.**—C. Chabré and A. Bouchonnet.—Of the four compounds of sulphur and iridium which have been described one only appears to have certain existence, the sesquisulphide. The authors, therefore, when investigating selenium compounds, endeavour to obtain the selenide corresponding to the sesquisulphide. This compound is prepared by a method analogous to that by which the sulphide is prepared; i.e., by passing a current of seleniuretted hydrogen into a solution of sesquichloride of iridium and slightly heating. The substance after purification is analysed, and is found to be similar in constitution and properties to the sesquisulphide.

**Acetates of the Alkaline Earths.**—Albert Colson.—To prepare the acetates of magnesium and calcium the author adds a sufficient quantity of acetic acid to the anhydride of the metal. In the case of magnesium the acetate  $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 1.5\text{C}_2\text{H}_4\text{O}_2$  is formed, identical with that formed by adding metallic magnesium to glacial acetic acid.

**Action of Bromosuccinic and Bibromosuccinic Acid on the Pyridic and Quinolic Bases.**—Louis Dubreuil.—The action of pyridic and quinolic bases on the bromine derivatives of succinic acid vary with the nature of the base and that of the solvent. According to the circumstances, malic, fumaric, bromofumaric, bromomaleic acid, or bicarbonic acetylene, are formed.

**A New Tri-iodine Phenol.**—P. Brenans.—In previous researches the author prepared the di-iodine isomers of phenol,  $\text{OH}-\text{C}_6\text{H}_3\text{I}_2 \cdot 1.2.4, 1.2.6, 1.3.6, 1.3.5$ , and  $1.3.4$ , as well as the nitro-benzenes and the iodine anilines from which they are prepared. He now describes the iodine compounds which are obtained from di-iodine orthonitroaniline,  $\text{NH}_2-\text{C}_6\text{H}_3\text{I}_2-\text{NO}_2 \cdot 1.4.6$ , by the following series of reactions:—1. The diazoic sulphate of this nitraniline is decomposed with potassium iodide, and changes into tri-iodine nitro-benzene,  $\text{NO}_2-\text{C}_6\text{H}_2\text{I}_3 \cdot 1.3.5.6$ . This nitrated derivative gives on reduction a tri-iodine aniline,  $\text{NH}_2-\text{C}_6\text{H}_2\text{I}_3 \cdot 1.3.5.6$ . This base is then diazotised, and the diazo compound heated in presence of water, giving tri-iodine phenol,  $\text{OH}-\text{C}_6\text{H}_2\text{I}_3 \cdot 1.3.5.6$ . The author describes the conditions under which these transformations take place and the properties of the new bodies.

**Iodides of Mercur-ammonium of the Primary and Tertiary Amines.**—Maurice François.—The iodides of mercur-ammonium derivatives of the amines have been

very little studied. The author's researches show that the iodides of mercur-ammonium derivatives of the primary amines form a series parallel to that of the derivatives of ammonia, in which, however, the hydrogen of the ammonium is replaced by mercury.

**Stereoisomerism in the Substituted Campho-carbonic Ethers and Methyl-homocamphoric Acid. Ethyl Camphocarboxylic Acid.**—J. Minguin.

**Etherification of Phosphoric Acid by Glycerin.**—P. Carré.—The author establishes the fact that phosphorus acid forms with glycerin three ethers in air or vacuo—(1) a monoether, ordinary glycerophosphoric acid, a mono-acid to helianthine and a di-acid to phthalein; (2) a di-ether, mono-acid to helianthine and to phthalein; (3) a tri-ether neutral to indicators.

*Bulletin de la Société Chimique de Paris.*

Series 3, Vol. xxix., No. 12.

**The Electrolysis of Alkaline Sulphides.**—André Brochet and Georges Ranson.—Already noticed.

**The Electrolysis of Alkaline-earthly Sulphides.**—André Brochet and Georges Ranson.—Already noticed.

**The Electrolysis of Sulphide of Barium, with a Diaphragm.**—André Brochet and Georges Ranson.—Already noticed.

**Amide and Nitride of Barium.**—MM. Guntz and Mentrel.—A fragment of barium placed in a U-tube through which a current of ammonia is passed, and plunged into a transparent melted bath of chloride of lithium and chloride of potassium in molecular proportions, does not show any change up to 280°. At this temperature, however, the metal becomes covered with a blackish liquid through which a large number of small bubbles of hydrogen pass. On raising the temperature the liquid becomes green, then red, and on cooling goes through these changes again in the inverse order. But a pure product cannot be obtained in this manner, as the liquid formed attacks the glass, and the tubes break before cooling. For this reason we used iron boats instead. These boats were at first heated in a glass tube over a naked flame, and similar phenomena were observed; but if, eventually, the temperature was raised to about 600°, still in a current of ammonia, the product solidifies, and the evolution of gas continues even when there is no longer any free barium left in the boat. These changes show that various reactions take place at different temperatures. To determine the nature of these changes, the authors have made a number of experiments at constant and known temperatures, using M. Guntz's electrical heating apparatus already described (*Bull. Soc. Chim.*, Series 3, vol. xxvii., p. 153). To prevent projections, and the consequent decomposition of the products on contact with the glass, the boats were surrounded with a thin sheet of iron or nickel. Experiment showed that at 280° hydrogen commenced to come off; the temperature can be raised to 380–400° without the liquid formed being decomposed on contact with the iron or nickel cover. On cooling, the mass appears as a greyish-white homogeneous substance, giving off ammonia in moist air. Three estimations of the total alkalinity and ammonia give:—

|                                    | I.   | II.   | III. | Calculated for<br>$\text{Ba}(\text{NH}_2)_2$ |
|------------------------------------|------|-------|------|----------------------------------------------|
| $\text{NH}_3$ /total alkalinity .. | 0.48 | 0.496 | 0.48 | 0.50                                         |

It must be remembered that these products contain all the impurities of the original barium. These figures correspond to the formula  $\text{Ba} + 2\text{NH}_3 = \text{Ba}(\text{NH}_2)_2 + \text{H}_2$ . This amide was obtained best by preventing the fused product from boiling. Further experiments were made on the decomposition of the amide in a current of ammonia gas, and in vacuo, when the nitride is formed.

**Some Amino-magnesian Phosphates; Methyl-amino- and Trimethyl-amino-magnesian Phosphates.**—Ch. Porcher and M. Brissac.—A solution of hydrochlorate of amine is mixed with a 10 or 15 per cent solu-



tion of disodic-phosphate in slight excess; on adding a solution of sulphate of magnesia, a slight crystalline cloudiness appears. This is dissolved by adding a few drops of hydrochloric acid and then the free base until strongly alkaline. Or the mixture of the hydrochlorate of the base and phosphate of soda can be made strongly alkaline, and the solution of sulphate of magnesia added gradually; the results obtained are the same whichever method is adopted. The precipitate obtained is left for twenty-four hours, filtered, washed carefully with water, alcohol, and ether, then drained and dried for a few minutes at 40°. This is the methylamino-magnesian phosphate. The trimethylamino-magnesian phosphate is prepared in a similar manner, using trimethylamine in the first instance.

Action of some Gases on Barium-ammonium.—MM. Guntz and Mentrel.—Inserted in full (p. 13).

Some Amino-magnesian Arseniates; Methylamino- and Trimethylamino-magnesian Arseniates.—M. Brissac.—The formation of the amino-magnesian arseniates corresponding to the above mentioned phosphates is brought about under the same conditions as for these latter, except that a greater alkalinity is necessary, and care must be taken not to use too much sulphate of magnesia.

Behaviour of Phenolphthalein in the presence of Bicarbonates and Alkaline Carbonates.—M. Giraud.—Inserted in full (p. 27).

The Use of Binoxide of Lead in Analysis.—M. St. Bogdan.—Inserted in full (p. 26).

Some Reactions of Pinacolin and of Pinacone.—G. Denigès.—The author considered that pinacolin, in its character of a ketone, ought to give with sulphate of mercury a compound analogous to that he has already described with ordinary acetone and its homologues. This was found to be the case; on heating a drop of pinacolin with 5 c.c. of acid sulphate of mercury in a glass tube in a boiling water-bath, after a few minutes a yellow colour was observed which soon gave place to a heavy yellow precipitate in spherocrystals, corresponding to the expected compound. Other experiments gave similar confirmatory results. The results prove the ketonic formula of pinacolin, and consequently of the other pinacolins so closely related to it; further, they enable us to detect pinacone itself with ease.

The Nitric Ethers of the Acid Alcohols.—H. Duval.—Twenty-five grms. of pure white glycolic acid are ground up rapidly in 30 grms. of nitric acid of 1.45 density, and while keeping cool, 25 grms. of concentrated sulphuric acid are added; let stand, and pour the whole over 100 grms. of crushed ice and 50 grms. of water. Then extract successively with 100 c.c. and 50 c.c. of ether, taking care to keep the temperature down to 0°. The ethereal solution is washed with water until all the sulphuric acid has disappeared, then evaporated; the product, dried over sulphate of soda, is taken up with anhydrous ether, filtered, and finally placed *in vacuo* over sulphuric acid, where it crystallises in forty-eight hours. The return is over 37 per cent. Its formula is  $\text{CH}_2\text{—ONO}_2\text{—CO}_2\text{H}$ .

A New Di-iodised Phenol.—P. Brenans.—Already noticed.

Migration of the Methyl Group in the Camphor Molecule.—G. Blanc and M. Desfontaines.—Already noticed.

Precipitation of some Alkaloids by Nitrate of Uranium. Reaction of Morphine.—J. Aloy.—Inserted in full (p. 26).

Influence of the Nature of its Surroundings on the Degree of Hydration of a Plant.—E. Charabot and A. Hébert.—Not suitable for abstraction.

Attempts to make Aniline and Urea take the Form of Amino- (Anilino- or Ureo-) Magnesian Phosphate.—Ch. Porcher and M. Brissac.—The authors have found that as they deal with fatty amines richer in carbon, the amino-magnesian phosphate obtained loses its stability,

or even does not form at all. In this manner the accumulation of carbon in the amine tends to diminish the basic character which is so marked in the first ones.

## MISCELLANEOUS.

Accidents in Tar Distilleries.—The attention of the Home Office has been directed by the reports of the Inspectors of Factories, and by the occurrence of fatal accidents, to the serious risks incurred by persons employed in works in which is carried on the distillation of tar for the production of naphtha, light oil, creasote, and pitch. It is thought desirable, pending a revision of the existing special rules for chemical works, to urge certain recommendations upon occupiers; these are for the most part based upon the precautions actually in use in certain works. The recommendations are nine in number, and are issued on "Form 919" from the Home Office, October, 1903. Instructions are also given for the resuscitation of persons who have been "gassed," and for the administration of oxygen.

Regulations for the Manufacture of Electric Accumulators.—These regulations were issued in draft by the Home Office on August 10, 1903, and no objection of substance (except those since withdrawn) was made by employers, by workmen, or by other persons affected. They have now been formally made by the Secretary of State, and issued in printed form, and will apply to such works on and after January 1, 1904. Besides these regulations, the Home Office has sent out a number of observations by way of explanation of certain of the regulations, and, further, to indicate additional safeguards which it has not been thought practicable to define as formal requirements. Copies of these regulations must be kept posted up in conspicuous places in all factories and workshops to which they apply. They can be obtained from Messrs. Eyre and Spottiswoode, East Harding Street, London, E.C.

## MEETINGS FOR THE WEEK.

TUESDAY, 19th.—Royal Institution, 5. "Development of Animals," by Prof. L. C. Miall, F.R.S.  
Society of Arts, 8. "Celtic Ornament," by George Coffey.

WEDNESDAY, 20th.—Microscopical, 8. Dr. Woodward's Presidential Address, "On the Evolution of Vertebrate Animals in Time."  
Society of Arts, 8. "Organ Design," by Thomas Cason.

Chemical, 5.30. "Optically Active Asymmetric Nitrogen Compounds. *d*- and *l*-Phenylmethyl-ethylmethylammonium Salts," by H. O. Jones.  
"Chemical Reactions of Nickel Carbonyl—Part I, Reactions with the Halogens, &c.; Part II, Reaction with Aromatic Hydrocarbons in presence of Aluminium Chloride, Synthesis of Aldehydes and Anthracene Derivatives," by J. Dewar and H. O. Jones.  
"A Microscopical Method of Determining Molecular Weights," by G. Barger.  
"o-Nitrobenzoyl-acetic Acid," by E. R. Needham and W. H. Perkin, jun.  
"The *cis* and *trans* Modifications of *o*-Trimethylglutaconic Acid," by W. H. Perkin, jun., and Miss A. E. Smith.  
"Influence of Nuclear Substitution on the Rate of Oxidation of the Side-chain—I, Oxidation of the Mono- and Dichlorotoluenes," by J. B. Cohen and J. Miller.  
"Simple Thermostat for Use in connection with the Refractometric Examination of Oils and Fats" and "The Interdependence of the Physical and Chemical Criteria in the Analysis of Butter-fat," by I. E. Thorpe.  
"Condensation of Furfuraldehyde with Sodium Succinate," by A. W. Titherley and J. F. Spencer.

THURSDAY, 21st.—Royal Institution, 5. "The Flora of the Ocean," by G. R. M. Murray, F.R.S.

FRIDAY, 22nd.—Royal Institution, 9. "Spectroscopic Studies of Astrophysical Problems at Stonyhurst College Observatory," by the Rev. Walter Sidgreaves.

SATURDAY, 23rd.—Royal Institution, 3. "British Folk Song" (with Vocal Illustrations), by J. A. Faller Maitland, M.A.

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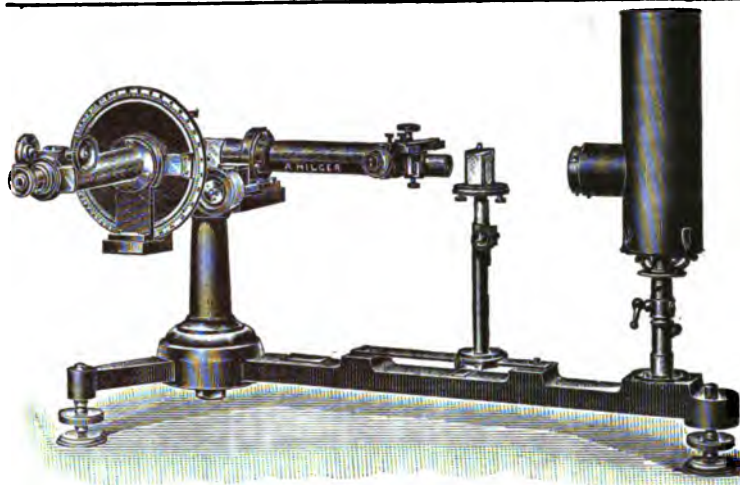
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### CONTENTS.

| ARTICLES:—                                                                                                               | PAGE |
|--------------------------------------------------------------------------------------------------------------------------|------|
| The Thermo-chemistry of the Theory of Electrolytic Dissociation, by Joseph W. Richards .....                             | 37   |
| Proposed Method of Testing Wood Treated to Resist Fire, by Chas. F. McKenna .....                                        | 40   |
| Report on Ash, by G. S. Fraps .....                                                                                      | 41   |
| Precipitation and Separation by Weak Organic Bases, by E. T. Allen .....                                                 | 43   |
| The Analysis of Commercial Nickel, by A. Hollard .....                                                                   | 45   |
| NOTICES OF BOOKS .....                                                                                                   | 46   |
| CORRESPONDENCE.—What may Result from the Study of Radium —Why are some Elements so much more Abundant than others? ..... | 47   |
| MISCELLANEOUS .....                                                                                                      | 47   |
| MEETINGS FOR THE WEEK .....                                                                                              | 48   |

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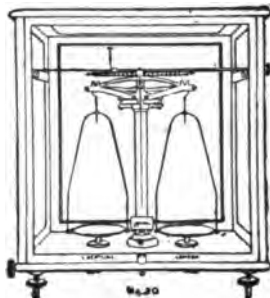
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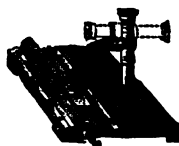
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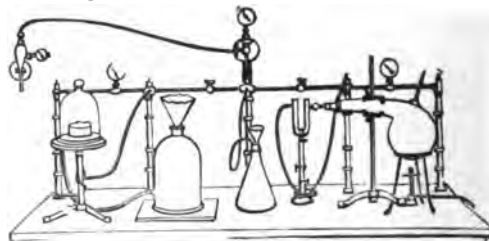
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2304.

## THE THERMO-CHEMISTRY OF THE THEORY OF ELECTROLYTIC DISSOCIATION.\*

By JOSEPH W. RICHARDS.

(Concluded from p. 32).

ASSUMING that the heat of formation of salts to dilute solutions represents the heat of formation of the ions from the elements, and further assuming as the basis of calculation the value of the mercury sulphate electrode - 0.89 volt, giving the thermo-chemical constant of mercury - 20,500 calories, we can construct the accompanying table of thermo-chemical constants, which shows the contribution which each base or acid radicle (forming from its elements) furnishes towards the heat of formation of the compound dissolved to dilute solution.

In using these data, the heat of formation of a compound in dilute solution, from its elements, is the sum of the thermochemical constants of its ions, each in dilute solution; the thermochemical constant per chemical equivalent of each ion, however, being multiplied first by the valence of the ion. If the ions, on electrolysis, appear at the electrodes as the elementary constituents of the compound, then the voltage necessary for decomposition is simply the sum of the thermochemical constants of the ions per chemical equivalent divided by 23.040. If the result of the passage of the current furnishes other products than those at the electrodes, then the heat of formation of these products must be allowed for, and subtracted from the heat of formation of the compound from its elements, in order to find the net work which the current then performs.

It must not be overlooked, either, that the basis or base line on which the table is constructed is an arbitrary one, corresponding to  $Hg = Hg' = -41,000$  calories, and that if this value should be really smaller (numerically greater), the thermochemical constants of all bases would be correspondingly decreased, while those of all acid ions would be correspondingly increased, and *vice versa*.

### Heat of Neutralisation.

To make immediate and specific application of the principles enunciated in the preceding part of this paper, let us consider the phenomenon of the heat of neutralisation of bases by acids, in dilute solution. Passing at once to a concrete illustration, we will take up Thomson's determination of the neutralisation of dilute caustic soda by dilute hydrochloric acid. The equation is—



The heat of neutralisation per chemical equivalent of the ions concerned is—

$$+13,740 \text{ calories at } 18^\circ C.$$

In the dilute solution of NaOH we have in the terms of the dissociation theory, ionised molecules of NaOH, viz., Na' and (OH)' ions; or we may make the statement broader, and simply say that we have molecules of NaOH in the condition called "ionised," by the dissociation theorists. From the standpoint of energetics, as explained in the preceding part of this paper, we have in that dilute solution simply combined Na and combined OH, each of these having contributed its thermochemical constant to the heat of formation of the compound. Similarly, in the dilute HCl, we have H' and Cl' ions, or, from the other

standpoint, combined H and combined Cl, forming or constituting the compound HCl.

If these two constituents could be mixed without neutralisation taking place, it will be generally admitted that the solution would contain ions of Na', (OH)', H', and Cl', or, in other words, combined Na, combined (OH), combined H, and combined Cl. From the standpoint of the dissociation theory, such a solution may equally well be regarded as composed of dissociated NaCl and HOH molecules, as of dissociated NaOH and HCl molecules; similarly, from the standpoint of energetics, it matters not which view we take of such a mixed solution; from this standpoint, it contains simply Na, OH, H, and Cl, all in the combined state.

If we now assume the neutralisation to take place in this mixed solution, what happens? The dissociation theory explains the action very simply by saying that since the resultant solution contains practically only Na and Cl ions, that the H and OH ions have united to form H<sub>2</sub>O, and that the heat of neutralisation is the heat of formation of H<sub>2</sub>O from H' and (OH)' ions, and is therefore a constant for any acid neutralising any base. To this statement, taken literally, I have very little objection, except to the term "heat of formation of water," as applied to the union of H' and (OH)' to form H<sub>2</sub>O.

The term "heat of formation of water" is a thermochemical term, implying that the constituents which form the water give up their thermochemical heats of combination and pass from the free state into the state of combination. But no such action, nor even a remotely analogous reaction, takes place when H ions unite with OH ions "to form water." The latter case is not one of thermochemical combination, for the H ions are already in the thermochemically combined state, as HCl, before the act of neutralisation, and the OH ions are similarly already combined as NaOH. It is then a fundamental mistake to regard the neutralisation as proceeding from an act of combination having the least analogy to a chemical act of combination, or of the neutralisation heat having the slightest analogy to thermochemical heat of combination. For, if the H and OH ions have once given out their thermochemical constants, while entering into the state of combination as HCl and NaOH, they cannot give them out again, or any part of them, in any further act of combination. The formation of liquid H<sub>2</sub>O from H and OH ions is therefore not a chemical act of combination, and the neutralisation heat is not in any sense thermochemical heat of combination. The heat of neutralisation, therefore, not resulting from chemical combination, must be physical in its nature.

The standpoint of energetics leads to the same view. Whatever the condition of molecules of HCl and NaOH in dilute solution may be, they are still compounds, and their constituents are all in the combined condition. After neutralisation, the Na and Cl are still in the combined condition, as NaCl, and the H and OH are still in the combined condition, as HOH. There has been therefore no change in the condition of any of the constituents named analogous to a change from the free to the combined condition, because they were all in the chemically combined condition before the act of neutralisation and all in the combined state afterwards. The heat evolved in neutralisation would be, therefore, as far as chemical combination occurs, zero, and its source must be a physical one.

The analogy between the molecular condition of a substance in the state of dilute solution, and the molecular condition characteristic of the gaseous state, furnishes the solution of the problem. We do not know exactly what the molecular structure of solids and liquids is, but it is essentially different from that of gases in the property of filling only a fixed maximum space, while gaseous molecules tend to occupy an indefinitely large space. Solid NaOH has a fixed minimum of specific gravity at its melting-point, where the molecules are as far apart as they can get from each other and the mass still retain the con-

\* A Paper read at the Fourth General Meeting of the American Electrochemical Society, at Niagara Falls, September 19, 1903.

## Thermo-chemical Constants of Bases.

|                                      |   | Per formula given.                      | Per chemical equivalent. |
|--------------------------------------|---|-----------------------------------------|--------------------------|
|                                      |   | Calories.                               | Calories.                |
| Li (solid) .. .. .                   | = | Li <sup>+</sup> .. .. .                 | + 62,000                 |
| K .. .. .                            | = | K <sup>+</sup> .. .. .                  | + 61,000                 |
| Na .. .. .                           | = | Na <sup>+</sup> .. .. .                 | + 56,300                 |
| Rb .. .. .                           | = | Rb <sup>+</sup> .. .. .                 | + 61,100                 |
| N + H <sub>4</sub> (gases) .. .. .   | = | (NH <sub>4</sub> ) <sup>+</sup> .. .. . | + 32,500                 |
| Sr (solid) .. .. .                   | = | Sr <sup>++</sup> .. .. .                | + 115,600                |
| Ca .. .. .                           | = | Ca <sup>++</sup> .. .. .                | + 107,000                |
| Mg .. .. .                           | = | Mg <sup>++</sup> .. .. .                | + 106,800                |
| Al .. .. .                           | = | Al <sup>+++</sup> .. .. .               | + 117,600                |
| Mn .. .. .                           | = | Mn <sup>+++</sup> .. .. .               | + 48,000                 |
| Zn .. .. .                           | = | Zn <sup>++</sup> .. .. .                | + 32,600                 |
| Fe .. .. .                           | = | Fe <sup>++</sup> .. .. .                | + 20,000                 |
| Fe <sup>+++</sup> (solution) .. .. . | = | Fe <sup>+++</sup> .. .. .               | - 12,100                 |
| Cd (solid) .. .. .                   | = | Cd <sup>++</sup> .. .. .                | + 16,200                 |
| Co .. .. .                           | = | Co <sup>++</sup> .. .. .                | + 14,600                 |
| Ni .. .. .                           | = | Ni <sup>++</sup> .. .. .                | + 13,600                 |
| Tl .. .. .                           | = | Tl <sup>+</sup> .. .. .                 | + 2,000                  |
| Sn .. .. .                           | = | Sn <sup>++</sup> .. .. .                | + 2,000                  |
| Pb .. .. .                           | = | Pb <sup>++</sup> .. .. .                | - 1,000                  |
| H <sub>2</sub> (gas) .. .. .         | = | 2H <sup>+</sup> .. .. .                 | - 1,800                  |
| Cu (solid) .. .. .                   | = | Cu <sup>++</sup> .. .. .                | - 17,600                 |
| Pt .. .. .                           | = | Pt <sup>+++</sup> .. .. .               | - 81,400                 |
| Hg (liquid) .. .. .                  | = | Hg <sup>++</sup> .. .. .                | - 41,000                 |
| Ag (solid) .. .. .                   | = | Ag <sup>+</sup> .. .. .                 | - 26,200                 |
| Au (solid) .. .. .                   | = | Au <sup>+++</sup> .. .. .               | - 93,600                 |

## Thermo-chemical Constants of Acid Elements.

|                      |    |    |    |    |   | Per reaction given. | Per chemical equivalent. |
|----------------------|----|----|----|----|---|---------------------|--------------------------|
|                      |    |    |    |    |   | Calories.           | Calories.                |
| O <sub>2</sub> (gas) | .. | .. | .. | .. | = | 2O'' + 86,400       | + 21,600                 |
| Cl <sub>2</sub> "    | .. | .. | .. | .. | = | 2Cl' + 80,600       | + 40,300                 |
| Br <sub>2</sub> "    | .. | .. | .. | .. | = | 2Br' + 66,400       | + 33,200                 |
| I <sub>2</sub> "     | .. | .. | .. | .. | = | 2I' + 28,200        | + 14,100                 |
| F <sub>2</sub> "     | .. | .. | .. | .. | = | 2F' + 107,600       | + 53,800                 |
| S (solid)            | .. | .. | .. | .. | = | S'' - 8,400         | - 4,200                  |
| Se (met.)            | .. | .. | .. | .. | = | Se'' - 34,000       | - 17,000                 |

dition of solid material; liquid NaOH has a fixed minimum of specific gravity, at its boiling-point, where the molecules are as far apart as they can get from each other and the mass still retain the condition of liquid material. If we force the material to occupy a larger space, its molecules pass into the condition characteristic of a gas.

Solution is a process by which the spatial relations of the molecules of the dissolved substance may be indefinitely extended, and by which, therefore, the substance takes on the molecular condition of the gaseous state. A cubic centimetre of solid NaCl dissolved in a cubic metre of water passes as certainly into the gaseous condition as if it had been vaporised, for the molecules of salt are further apart in this dilute solution than they would be in vapour of NaCl. I need scarcely take the time to prove this proposition arithmetically, since this view of the subject is qualitatively and quantitatively proven by the phenomena of osmotic pressure. The facts of osmotic pressure prove that a substance in dilute solution exerts pressure which has all the characteristics of gas pressure, and agrees quantitatively with the pressure which the material could exert in the state of gas if occupying the volume of the solution, but in the gaseous state. The temperature coefficient of the osmotic pressure, moreover, agrees with the gas coefficient. I believe that all will agree, at least, on the very close resemblance or analogy between the gaseous state and the state of being in dilute solution, and a consideration of the heat of neutralisation from the point of view taken in this paper tends to confirm their practical equivalence.

Since the molecule of NaOH in dilute solution exerts osmotic pressure, analogous to its being in the gaseous condition, and the same is true of the molecule of HCl, the two solutions, if mixed without neutralisation taking place, would exert in the mixed condition osmotic pressure as in

each solution separately, all the constituents being in the "ionised" condition and exerting osmotic pressure analogous to gaseous pressure. When neutralisation takes place, only Na and Cl are left in dilute solution, and exert the osmotic pressure, the pressure due to the H and OH has disappeared, coincident with these passing into the condition of liquid water. The act of neutralisation is therefore merely the passage of H and OH from the combined state in dilute solution, in which condition they exert gaseous, osmotic pressure, into the combined state as liquid water, in which they exert practically no osmotic pressure. The change is exactly analogous to the change from the gaseous into the liquid condition, and is therefore the equivalent of the condensation of water vapour into the liquid state. The source of the heat of neutralisation is therefore the physical change of state of HOH from the gaseous to the liquid condition, without any heat of chemical combination being concerned or even possible, because the ingredients, H and OH, are as much in the combined condition before the act of neutralisation as after it.

To verify this statement quantitatively, we need to take into consideration the fact that 2NaOH.400H<sub>2</sub>O will occupy 7.2 litres (if the molecular weights be expressed in grammes), and the NaOH will exert an osmotic pressure of 12.34 atmospheres. The 2HCl.400H<sub>2</sub>O occupies an equal volume, and the HCl exerts a similar osmotic pressure. After the mixing of the solutions the osmotic pressure exerted by each molecule in solution is halved, but the total osmotic pressure is 12.34 atmospheres. After neutralisation, there remains only the osmotic pressure of the 2NaCl in the 802H<sub>2</sub>O, which is 6.15 atmospheres, the osmotic pressure of the H and OH together equal to 6.19 atmospheres, disappearing because of the condensation of the water to the liquid state. The heat given out in



Thermo-chemical Constants of Acid Radicals.

|                         |                           |                             |                                                   | Per reaction<br>given.<br>Calories. | Per chemical<br>equivalent.<br>Calories. | Salt formed.      |
|-------------------------|---------------------------|-----------------------------|---------------------------------------------------|-------------------------------------|------------------------------------------|-------------------|
|                         | O <sub>2</sub> (gas) plus | H <sub>2</sub> (gas) equals | 2(OH)'                                            | + 112,200                           | + 56,100                                 | Hydrate.          |
|                         | S <sub>2</sub> (solid) "  | H <sub>2</sub> " "          | 2(SH)'                                            | + 8,600                             | + 4,300                                  | Sulphhydrate.     |
|                         | Se <sub>2</sub> (met.) "  | H <sub>2</sub> " "          | 2(SeH)'                                           | + 40,000                            | + 20,000                                 | Selenhydrate.     |
|                         | Cl <sub>2</sub> (gas) "   | O <sub>2</sub> " "          | 2(ClO)'                                           | + 56,800                            | + 28,400                                 | Hypochlorite.     |
|                         | Cl <sub>2</sub> " "       | 3O <sub>2</sub> " "         | 2(ClO <sub>3</sub> )'                             | + 45,600                            | + 22,800                                 | Chlorate.         |
|                         | Cl <sub>2</sub> " "       | 4O <sub>2</sub> " "         | 2(ClO <sub>4</sub> )'                             | + 80,600                            | + 40,300                                 | Perchlorate.      |
|                         | Br <sub>2</sub> " "       | O <sub>2</sub> " "          | 2(BrO)'                                           | + 59,000                            | + 29,500                                 | Hypobromite.      |
|                         | Br <sub>2</sub> " "       | 3O <sub>2</sub> " "         | 2(BrO <sub>3</sub> )'                             | + 26,800                            | + 13,400                                 | Bromate.          |
|                         | I <sub>2</sub> " "        | 3O <sub>2</sub> " "         | 2(IO <sub>3</sub> )'                              | + 131,800                           | + 65,900                                 | Iodate.           |
|                         | I <sub>2</sub> " "        | 4O <sub>2</sub> " "         | 2(IO <sub>4</sub> )'                              | + 107,000                           | + 53,500                                 | Periodate.        |
|                         | 4S (solid)                | 3O <sub>2</sub> " "         | 2(S <sub>2</sub> O <sub>3</sub> )''               | + 290,600                           | + 72,650                                 | Hyposulphite.     |
|                         | 2S " "                    | 3O <sub>2</sub> " "         | 2(SO <sub>3</sub> )''                             | + 304,000                           | + 76,000                                 | Sulphite.         |
| 2S (solid) +            | 3O <sub>2</sub> (gas)     | H <sub>2</sub> " "          | 2(HSO <sub>3</sub> )'                             | + 300,600                           | + 150,300                                | Bisulphite.       |
|                         | 4S (solid)                | 5O <sub>2</sub> " "         | 2(S <sub>2</sub> O <sub>5</sub> )''               | + 464,400                           | + 116,100                                | Pyrosulphite.     |
|                         | S " "                     | 2O <sub>2</sub> " "         | (SO <sub>4</sub> )''                              | + 215,800                           | + 107,900                                | Sulphate.         |
| H <sub>2</sub> (gas) +  | 2S " "                    | 4O <sub>2</sub> " "         | 2(HSO <sub>4</sub> )'                             | + 424,000                           | + 212,000                                | Bisulphate.       |
|                         | 2S " "                    | 4O <sub>2</sub> " "         | (S <sub>2</sub> O <sub>8</sub> )''                | + 318,000                           | + 159,000                                | Persulphate.      |
|                         | 3S " "                    | 3O <sub>2</sub> " "         | (S <sub>2</sub> O <sub>6</sub> )''                | + 278,500                           | + 139,250                                | Dithionate.       |
|                         | 3S " "                    | 3O <sub>2</sub> " "         | (S <sub>3</sub> O <sub>6</sub> )''                | + 274,800                           | + 137,400                                | Trithionate.      |
|                         | 4S " "                    | 3O <sub>2</sub> " "         | (S <sub>4</sub> O <sub>6</sub> )''                | + 263,000                           | + 131,500                                | Tetrathionate.    |
|                         | 5S " "                    | 3O <sub>2</sub> " "         | (S <sub>5</sub> O <sub>6</sub> )''                | + 268,000                           | + 134,000                                | Pentathionate.    |
|                         | 2Se " "                   | 3O <sub>2</sub> " "         | 2(SeO <sub>3</sub> )''                            | + 243,800                           | + 60,950                                 | Selenite.         |
|                         | Se " "                    | 2O <sub>2</sub> " "         | (SeO <sub>4</sub> )''                             | + 147,400                           | + 73,700                                 | Selenate.         |
|                         | N <sub>2</sub> (gas) "    | O <sub>2</sub> " "          | 2(NO)'                                            | - 5,800                             | - 2,900                                  | Hyponitrite.      |
|                         | N <sub>2</sub> " "        | 2O <sub>2</sub> " "         | 2(NO <sub>2</sub> )'                              | + 55,800                            | + 27,900                                 | Nitrite.          |
|                         | N <sub>2</sub> " "        | 3O <sub>2</sub> " "         | 2(NO <sub>3</sub> )'                              | + 99,400                            | + 49,700                                 | Nitrate.          |
|                         | P (solid)                 | 2O <sub>2</sub> " "         | (PO <sub>4</sub> )'''                             | + 300,600                           | + 100,200                                | Phosphate.        |
| H <sub>2</sub> (gas) +  | 2P " "                    | 4O <sub>2</sub> " "         | 2(HPO <sub>4</sub> )''                            | + 614,600                           | + 153,650                                | Mono-H-phosphate. |
| H <sub>2</sub> (gas) +  | P " "                     | 2O <sub>2</sub> " "         | (H <sub>2</sub> PO <sub>4</sub> )''               | + 308,600                           | + 308,600                                | Di-H-phosphate.   |
|                         | As " "                    | O <sub>2</sub> " "          | (AsO <sub>2</sub> )''                             | + 206,100                           | + 103,050                                | Arsenite.         |
|                         | As " "                    | 2O <sub>2</sub> " "         | (AsO <sub>4</sub> )'''                            | + 213,300                           | + 71,100                                 | Arsenate.         |
| H <sub>2</sub> (gas) +  | 2As " "                   | 4O <sub>2</sub> " "         | 2(HAsO <sub>4</sub> )''                           | + 435,800                           | + 72,600                                 | Mono-H-arsenate.  |
| H <sub>2</sub> (gas) +  | As " "                    | 2O <sub>2</sub> " "         | (H <sub>2</sub> AsO <sub>4</sub> )''              | + 218,100                           | + 218,100                                | Di-H-arsenate.    |
|                         | 2C (amorph.)              | N <sub>2</sub> " "          | 2(CN)'                                            | - 68,000                            | - 34,000                                 | Cyanide.          |
| O <sub>2</sub> (gas) +  | 2C " "                    | N <sub>2</sub> " "          | 2(CNO)'                                           | + 76,000                            | + 38,000                                 | Cyanate.          |
| 2S (solid) +            | 2C " "                    | N <sub>2</sub> " "          | 2(CNS)'                                           | - 34,400                            | - 17,200                                 | Sulphocyanate.    |
| Fe (solid) +            | 6C " "                    | 3N <sub>2</sub> " "         | (FeC <sub>6</sub> N <sub>6</sub> )'''             | - 98,800                            | - 24,700                                 | Ferrocyanide.     |
| Fe (solid) +            | 6C " "                    | 3N <sub>2</sub> " "         | (FeC <sub>6</sub> N <sub>6</sub> )'''             | - 155,800                           | - 51,900                                 | Ferricyanide.     |
|                         | 2C " "                    | 3O <sub>2</sub> " "         | 2(CO <sub>3</sub> )''                             | + 333,400                           | + 83,350                                 | Carbonate.        |
| H <sub>2</sub> (gas) +  | 2C " "                    | 3O <sub>2</sub> " "         | 2(HCO <sub>3</sub> )'                             | + 340,000                           | + 170,000                                | Bicarbonate.      |
| H <sub>2</sub> (gas) +  | 2C " "                    | 2O <sub>2</sub> " "         | 2(CHO <sub>2</sub> )'                             | + 211,000                           | + 105,500                                | Formate.          |
| 3H <sub>2</sub> (gas) + | 4C " "                    | 2O <sub>2</sub> " "         | 2(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )' | + 242,800                           | + 121,400                                | Acetate.          |
|                         | 2C " "                    | 2O <sub>2</sub> " "         | (C <sub>2</sub> O <sub>4</sub> )''                | + 201,400                           | + 100,700                                | Oxalate.          |

this condensation is equal numerically to that necessary to convert 2H<sub>2</sub>O in the liquid state into vapour exerting in the gaseous state a pressure of 6.19 atmospheres.

The latent heat of vaporisation of 1 kilo. of H<sub>2</sub>O at 0° is 606.5 calories (Regnault), or for 18 parts 10.917 calories. If we subtract the outer work done in giving tension to the vapour (2T) the work of vaporisation without outer work is 10.917 - 546 = 10.371 calories. At 18° by a similar calculation the work of vaporisation is—

$$(594 \times 18) - 2(273 + 18) = 10,692 - 582 = 10,110 \text{ calories.}$$

The outer work, however, if the vapour exerts 6.19 atmospheres pressure, is 6.19 × 2T = 6.19 × 582 = 3590 calories. The total work of vaporisation, including the work necessary to give the vapour the requisite tension of 6.19 atmospheres is 10,110 + 3590 = 13,700 calories. Therefore, combined H and OH, at a tension of 6.19 atmospheres, will give out in condensing to combined H and OH, as liquid water, at 18° C., 13,700 calories. The heat of neutralisation, 13,740 calories, is, therefore, nothing else than the physical heat of condensation of 18 parts of combined H and OH to 18 parts of liquid water.

An exactly similar conclusion follows from a thermo-chemical consideration of the heat of formation of an "ionised" H<sub>2</sub>O molecule in dilute solution. The thermochemical constants of H' and (OH)' are already given in the tables, and their sum must be the heat of formation of water from hydrogen and oxygen gases,

when the molecule remains in dilute solution, i.e., "ionised."

$$\begin{aligned} \text{H}' &= -900 \text{ calories} \\ (\text{OH})' &= +56,100 \text{ " } \\ \text{Sum} &= +55,200 \text{ " } \end{aligned}$$

This represents the heat of formation in dilute solution, with the molecule "ionised," so as to exert the usual osmotic pressures of H and OH ions in the dilute solutions used. In the concrete case of mixed NaOH and HCl solutions, we have found this osmotic pressure to have been 6.17 atmospheres. To calculate what the heat of formation of the "ionised" molecule would be if it exerted 1 atmosphere pressure, we add to 55,200 the work of expansion from 6.19 atmospheres to 1 atmosphere = 6.19 - 1 × 2T = 5.19 × 582 = 3021 calories, giving a total of 58,221 calories. But the heat of formation of vapour of water at 18° from oxygen and hydrogen gas is, as nearly as can be calculated from Regnault's experiments, 58,100 calories. It thence follows, that the heat of combination of hydrogen and oxygen gases to form an "ionised" molecule, in dilute solution, the ionised molecule exerting an osmotic pressure of one atmosphere, is equal to or identical with the heat of combination to form vapour of water at one atmosphere tension; from which follows the substantial identification of the "ionised" water molecule H' and (OH)' ions at a

given osmotic pressure) with the gaseous state of water vapour at the same pressure.

Two lines of reasoning and calculation therefore give proof to the thesis, that the heat of neutralisation is merely the heat evolved in the condensation of a gaseous  $H_2O$  molecule into the liquid state, and this conclusion, again, agrees further with the conception that the "ionised" condition of a molecule in dilute solution is merely a certain physical condition of the compound, and that "ionisation" is in no sense to be regarded as a resolution or decomposition of the molecule into its constituents. An "ionised" molecule is still a molecule of the compound, with its constituents still in the combined condition.

If it be objected that the heat of neutralisation is not a constant for all acids and bases, but that there are irregularities, it has been shown by Arrhenius that these irregularities are due to imperfect or incomplete "ionisation" of either the acid or base or both, and that when this is allowed for, the irregularities disappear or are explained. Exactly the same line of reasoning would account for the irregularities, assuming the line of explanation of the heat of neutralisation which has been brought forward in this paper.

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## A PROPOSED METHOD OF TESTING WOOD TREATED TO RESIST FIRE.\*

By CHAS. F. McKENNA

(Concluded from p. 33).

THE following experiments demonstrate that the gas yield and coal from the distillation of small pieces of some of the more common woods could be used as a criterion of the extent of retardation of fire caused by the proofing processes.

Spruce; 0.500 grm.; fireproof; heated for two minutes by 9 ampere current:—

|           |       |            |
|-----------|-------|------------|
| Gas yield | .. .. | 116.5 c.c. |
| " "       | .. .. | 124.5      |
| " "       | .. .. | 117.0      |
| Average   | .. .. | 119.3      |

The same wood leached in boiling water and dried to normal moisture:—

|           |       |            |
|-----------|-------|------------|
| Gas yield | .. .. | 136.5 c.c. |
| " "       | .. .. | 129.0      |
| " "       | .. .. | 136.0      |
| Average   | .. .. | 133.8      |

Birch; 0.500 grm.; 8 ampere current for two minutes:—

| Gas Yield. |          |
|------------|----------|
| Untreated. | Treated  |
| 164 c.c.   | 151 c.c. |
| 165        | 160      |
| 171        | 162      |
| 165        | 153      |
| 167        | —        |
| Average .. | 166.4    |
|            | 156.5    |

The four specimens of char of the treated wood showed an average weight of 147.6 mg., and those of the untreated wood averaged 100 mg. The percentage of charcoal left in the treated wood was 29.5 per cent, and in the other 20 per cent.

With exercise of more care, similar experiments gave the following results:—

Birch; 0.500 grm.; 7 ampere current, 125 volts, two minutes:—

| Gas Yield. |            |
|------------|------------|
| Untreated. | Treated.   |
| 125 c.c.   | 95 c.c.    |
| 127        | 105        |
| 124        | 105        |
| 126        | 103        |
| 126        | 108        |
| 132        | 104        |
| Average .. | 126.6      |
|            | 103.3      |
| Charcoal.  |            |
| Untreated. | Treated.   |
| 0.1037 gr. | 0.1643 gr. |
| 0.1026     | 0.1540     |
| 0.1065     | 0.1595     |
| 0.1025     | 0.1700     |
| 0.1062     | 0.1681     |
| 0.1016     | 0.1661     |
| Average .. | 0.03541    |
|            | 0.16366    |

The following experiment was performed with the assistance of one of the companies practising the fire-proofing art: A sample of birch, of which I was given an initial untreated specimen, was fireproofed for me in a small test cylinder, using a solution of the density with which they were accustomed to operate on a large scale. Two solutions of less density by one-third and two-thirds of the dissolved salts were also prepared and used on specimens of the same wood on separate runs of the cylinder. I was thus provided with four specimens of wood of the same lot: the first, natural; the second, treated with a dilute solution; the third, with one more dense; and the fourth, with once most dense. These were numbered "0," "1," "2," and "3," respectively. In the test retort they gave the following results:—

0.500 grm.; 7 ampere current, 125 volts, two minutes:—

| Gas Yield.    |         |         |         |
|---------------|---------|---------|---------|
| "0"           | "1"     | "2"     | "3"     |
| 112 c.c.      | 89 c.c. | 99 c.c. | 97 c.c. |
| 108           | 92      | 97      | 95      |
| 104           | 88      | 92      | 92      |
| 107           | 87      | 99      | 93      |
| 107           | 89      | 95      | 91      |
| Average 107.6 | 88.6    | 96.4    | 93.6    |

Average weight of the charcoal from each:—

|     | Milligrams. | Per cent. |
|-----|-------------|-----------|
| "0" | .. .. 103   | 20.6      |
| "1" | .. .. 136   | 27.2      |
| "2" | .. .. 120   | 24.0      |
| "3" | .. .. 133   | 26.6      |

If the decomposition into gaseous products and coal measures the value of the treatment, the least dense solution gave the best result, which is probably true, as it was an alum process, and the penetration of the cell-walls was probably more perfect with the most dilute solution.

Experiments with Yellow Pine, treated by another process and patent than the preceding. This yellow pine gave the following results (as there was no original stock offered, the "untreated" specimens were obtained from scraps of yellow pine in a carpenter shop):—

0.500 grm.; 8 ampere current, 125 volts, two minutes:—

| Gas Yield. |          |
|------------|----------|
| Untreated. | Treated. |
| 125 c.c.   | 87 c.c.  |
| 131        | 86       |
| 128        | 88       |
| 133        | 86       |
| 134        | 86       |
| Average .. | 130.2    |
|            | 86.6     |

\* From the *Journal of the American Chemical Society*, xxv., No. 4.

Average weight of four samples of charcoal :—

Untreated . . . 0.0979 grm. or 19.58 per cent.  
Treated . . . 0.1912 grm. or 38.24 per cent.

Yellow pine untreated :—

- (a) Dried.  
(b) With normal moisture, 7.1 per cent more water than (a).

Gas Yield.

| (a)            | (b)      |
|----------------|----------|
| 139 c.c.       | 128 c.c. |
| 136            | 132      |
| 143            | 134      |
| Average .. 136 | 131      |

Charcoal, average weight :—

| (a)        | (b)         |
|------------|-------------|
| 0.102 grm. | 0.0963 grm. |

Yellow pine, treated :—

- (c) Dried.  
(d) With normal moisture, 3.6 per cent more water than (c).

Gas Yield.

| (c)           | (d)     |
|---------------|---------|
| 90 c.c.       | 92 c.c. |
| 91            | 88      |
| 89            | 88      |
| 90            | 85      |
| Average .. 90 | 88      |

Charcoal, average weight :

| (c)        | (d)         |
|------------|-------------|
| 0.198 grm. | 0.1882 grm. |

Three questions arise :—Will not errors be introduced by the moisture of the wood? This is to be met in the same way as in previous methods of test; namely, by the preparation of dried samples subsequently exposed for the absorption of the normal amount of water. Whether it would vitiate the results obtained by inspectors having limited facilities for such work will have to be determined. Are not the results modified by the superior density of the treated sample over the normal wood that contains no salts? Naturally this should be so in finding the weight of the char residue; but in all experiments it seems to be an unimportant quantity, and in some it would appear that the wood which is heavier from treatment with the dense solution of an inferior fire-proofing salt will give a very large gas yield. Would not gases generated from the chemicals also modify the results? The only process where it possibly would be in using ammonium salts or carbonates, and proper provision should be made for the absorption of the gases as each case would require.

It would seem to me to be indicated by the preceding that the degree to which total decomposition without air can be inhibited by previous chemical treatment in wood subjected to a high heat can be definitely measured by subjecting small test pieces to distillation, that this test operation can be easily, quickly, and accurately performed in the small retort as shown, and that the number of such tests which can be made in a short time on small pieces is so great that a good average could be selected in chips from different parts, interior, exterior, &c., of lots of timber as delivered. It seems probable also that in the conditions surrounding experiments in such a retort, an equally accurate measure can be found of the resistance to inflammation of woods before and after treatment, with access of air.

I hope to continue such experiments in the future, if circumstances favour.

REPORT ON ASH.\*

By G. S. FRAPS.

BEFORE sending out samples or requests for co-operation the referee undertook the following work on the determination of sulphur :—

PRELIMINARY WORK ON SULPHUR.

The following methods were studied :—

1. Nitric-acid Method (the Provisional Method of the Association).

Five grms. of material are placed in a 3½-inch porcelain evaporating dish, 20 c.c. of nitric acid (concentrated) added, and the mixture heated cautiously on the water-bath until all danger of overflowing has passed. It is then partly evaporated, 10 c.c. of a 5 per cent solution of potassium nitrate is added, the mixture evaporated to complete dryness, and ignited, at first gently, then under a blast-lamp until the residue is white. It is then dissolved in hydrochloric acid, evaporated to dryness, and heated for some time in an air-bath to render silica insoluble. The residue is taken up in water with the addition of a little acid, filtered, and the sulphuric acid precipitated with barium chloride in the usual way.

This method gives good results. It has a disadvantage, however, in that the ash produced is easily fusible, acts upon the porcelain dish, and is ignited free of carbon with great difficulty. It was to overcome this objection that the modification described below was tested.

2. Nitric Acid Method Modified.

The modification consists in the use of 20 c.c. of a solution of calcium acetate containing 0.56 grm. of calcium oxide (CaO), in place of 20 c.c. of potassium nitrate solution. The ignition takes place much more easily, the ash does not fuse, and is more easily burned free of carbon than when potassium nitrate is used.

3. Boiling with Nitric Acid.

An attempt was made to destroy the organic matter by boiling the material with nitric acid, which I understand is the method used for meats in the nutrition investigations of the Office of Experiment Stations. The method did not succeed with vegetable materials. Several experiments were made, but in no case was all organic matter of the vegetable materials destroyed, even when 5 grms. of material was boiled to dryness with 100 c.c. nitric acid in a Kjeldahl flask. As it would therefore be necessary to wash the residue from the flask into a dish, and ignite with the addition of potassium nitrate or calcium acetate to remove organic matter, this method would differ from the two preceding only in that a larger quantity of acid is used and the digestion with the acid is more prolonged. The method was not further examined.

4. Fusion with Caustic Soda.

This is the standard method for determining sulphur in non-volatile organic compounds. The method as tested was as follows :—

Five grms. of material in a nickel crucible were saturated with 40 c.c. of a solution of 150 grms. sodium hydroxide in 300 c.c. water, evaporated to dryness, and fused with the addition of a little sodium peroxide. The fusion requires care to prevent the material from overflowing the dish, and the whole process is somewhat long. The melt was dissolved in water, neutralised, and acidified with hydrochloric acid, evaporated to dryness, and heated to render silica insoluble, filtered, and the sulphuric acid precipitated from the filtrate, the volume of which was about 200 c.c. The sodium hydroxide and sodium peroxide contained a large quantity of sulphates; about 0.1 grm. in the quantity used in the determination was found in the blank determination.

\* Proceedings of the Nineteenth Annual Convention of the Association of Official Agricultural Chemists, held at Washington, D.C.

The results with this method are decidedly low. This may possibly be due to the solubility of barium sulphate in sodium chloride, about 38 grms. of the latter salt being present in the solution from which the barium sulphate was precipitated, or it may be owing to the error caused by impurities in the reagents.

#### 5. Eschka's Method.

This is the method usually employed for the determination of sulphur in coals. It was applied to plant materials as follows:—

Five grms. material were intimately mixed in a platinum dish, with 71 grms. of a mixture of two parts magnesium oxide to one of dried sodium carbonate. The mixture was heated, stirring frequently, and the temperature raised slowly until all carbon appeared to be burned off. The mass was transferred to a beaker with about 50 c.c. water; 15 c.c. of saturated bromine water was added, and boiled five minutes. It was allowed to settle, decanted through a filter, boiled a second and third time with about 30 c.c. water, and washed thoroughly. The filtrate was made acid with hydrochloric acid, boiled until bromine was expelled, and then precipitated with barium chloride in the usual way. The results by this method were too low.

#### Results of Work.

The analytical results by the different methods are given in the following table (Table I.). The figures in each case are corrected for the sulphur in the reagents found by a blank determination. The blank was very low in the case of the two nitric acid methods. In the Eschka method it was equivalent to 0.0058 gm. barium sulphate in the quantity of the reagents used for each test. As has already been stated, the fusion method gave results much too low, and the blank was high, being 0.1103 gm. barium sulphate.

TABLE I.—Determination of Sulphur (SO<sub>3</sub>) by Different Methods.

|              | Nitric acid<br>method.<br>Per cent. | Nitric acid method<br>modified.<br>Per cent. | Eschka<br>method.<br>Per cent. |
|--------------|-------------------------------------|----------------------------------------------|--------------------------------|
| Corn .. ..   | 0.249<br>0.244                      | 0.287<br>0.286                               | 0.238<br>0.208                 |
| Average ..   | 0.247                               | 0.287                                        | 0.223                          |
| Peas .. ..   | 0.481<br>0.533<br>0.503             | 0.480<br>—<br>—                              | 0.297<br>0.312<br>0.313        |
| Average ..   | 0.518                               | 0.480                                        | 0.307                          |
| Millet .. .. | 0.369<br>0.376<br>0.400             | 0.427<br>0.434<br>—                          | 0.451<br>0.460<br>—            |
| Average ..   | 0.382                               | 0.431                                        | 0.456                          |

#### Official Work on Sulphur.

It was decided to compare the provisional method for the determination of sulphur with the method modified by the use of calcium acetate in place of potassium nitrate. A comparison between two methods for potash was also determined. The following circular letter was sent out to a number of members of this body:—

Raleigh, N.C., May 1, 1902.

DEAR SIR,—The referee on ash for the present year will continue the study of the methods of determining sulphur and potash in plant materials. It is thought best to work out one portion of the problem at a time. Kindly inform the referee if you will be able to co-operate. The samples will be sent out about the latter part of May. The work of last year proves conclusively that the sulphur in an ash, as ordinarily prepared, is no indication of the amount of sulphur in the plant, which

may be vastly greater. Kindly give us the benefit of any suggestions.—Very truly,

G. S. FRAPS, Referee.

FRANK T. SHUTT, Associate Referee.

Eight chemists requested samples. R. J. Davidson, of Virginia, sent the following table of analyses:—

TABLE II.—Determination of Sulphur (SO<sub>3</sub>) by Different Methods (Davidson).

| Method.                       | Dried apples,<br>sample 1.<br>Per cent. | Dried apples,<br>sample 2.<br>Per cent. | Bran.<br>Per cent. |
|-------------------------------|-----------------------------------------|-----------------------------------------|--------------------|
| Ignition with calcium acetate | 0.09                                    | 0.10                                    | 0.27               |
| Nitric acid method .. ..      | 0.09                                    | 0.10                                    | 0.45               |
| Eschka's method.. ..          | 0.10                                    | 0.11                                    | —                  |
| In ash .. ..                  | —                                       | —                                       | 0.09               |

W. P. Headen, of Fort Collins, Colo., wrote as follows:—

If you will refer to my Bulletin on Alfalfa, No. 35, of this station, pages 84—87, you will see that I did some work in this line in 1895 in regard to chlorine, sulphur, and phosphorus, but did nothing in regard to loss of potash. I found a very considerable loss of chlorine and sulphur, but none for phosphoric acid. I was rather surprised at the latter, as we know that phosphates heated with carbon, especially if there is sand enough present to set phosphoric acid free, may be reduced. In my experiments there were doubtless enough basic salts present to avoid such reactions; besides, the temperature of incineration was very low—as low as would accomplish the incineration.

You state that the sulphur in the plant may be vastly greater than that indicated by the determination of sulphuric acid or total sulphur in the ash. You will see that I found in one instance 4.88 per cent in the ash and 8.09 per cent in the plant, almost 66 per cent more.

Mr. Charles P. Beistle wrote as follows:—

I would say that I have done considerable work on the determination of sulphur in plant substances and proteid bodies, and have found the only satisfactory way is by fusion in a mixture of sodium hydrate and potassium nitrate. Among other methods used were ignition in a closed vessel containing oxygen under about 20 atmospheres pressure, ignition in a combustion tube in a current of oxygen gas, passing products of combustion through bromine water, and digestion in nitro-hydrochloric acid. The first mentioned method always gave the highest results. The next two methods gave almost as high results, but required special apparatus and a great expenditure of time. To determine sulphur in the ash of plants gives no indication whatever of the amount of sulphur present in the original material, and the calcium acetate method (simple ignition with calcium acetate), although giving higher results, is of no real value as a method of estimating total sulphur.

I would suggest that fusion with sodium peroxide might be successfully used as a method of oxidising the plant material and changing sulphur to sulphate.

#### Work on Ash.

Two samples were prepared, corn bran and cotton-seed meal, and sent out with the following letter:—

#### Directions for Work.

The samples consist of corn bran and cotton-seed meal. Determine moisture (in the usual way), sulphur, and potash in both samples. Make at least two determinations by each method. In reporting give weights of precipitates, per cent on samples as received, and on dry matter. Please report results as early as possible.

#### METHOD I.—Sulphur.

Place 5 grms. material in a 2½-inch porcelain evaporating dish, add 20 c.c. concentrated nitric acid, and heat the mixture cautiously on the water-bath until all danger of overflowing has passed. Then evaporate partly, add 10 c.c. of 5 per cent solution of potassium nitrate, evaporate to complete

dryness, and ignite, at first gently, then more vigorously until the residue is white. The residue is then dissolved in hydrochloric acid, evaporate to dryness, and heated for some time in an air-bath to render silica insoluble. The residue is taken up in water with the addition of a little hydrochloric acid, filtered to 150 c.c. or more, and the sulphuric acid precipitated with barium chloride in the usual way. Make a blank test with the reagents.

#### METHOD II.—Sulphur.

The same as the preceding, substituting 20 c.c. of a solution of calcium acetate for the potassium nitrate. The solution is prepared by dissolving 12.5 grms. calcium carbonate, chemically pure, in chemically pure acetic acid, and diluting to 250 c.c. Make a blank test with the reagents.

#### METHOD III.—Potash.

Determine by ignition with sulphuric acid as in the methods of the Association of Official Agricultural Chemists, page 22b. With the bran use a quantity of the solution corresponding to at least 2 grms. of bran.

#### METHOD IV.—Potash.

Char 10 grms. substance in a platinum dish, extract twice with hot dilute hydrochloric acid 1:10, and wash, receiving the extract and washing in a 500 c.c. flask. Return filter and contents to the dish, and ignite to whiteness. Dissolve the residue in hydrochloric acid, add to the extract in the flask, make up to volume, and determine potash as in Method III.

Report potash as  $K_2O$ , sulphur as  $SO_3$ . Please give the referee the benefit of your suggestions in regard to the work.

#### Moisture.

The moisture determinations on the samples sent out are given simply as a matter of record (Table III.). The agreement is satisfactory.

TABLE III.—Moisture Determinations.

| Analyst.                        | Corn bran. |  | Cotton-seed meal. |  |
|---------------------------------|------------|--|-------------------|--|
|                                 | Per cent.  |  | Per cent.         |  |
| J. H. Gibboney, Virginia .. ..  | 12.10      |  | 7.28              |  |
| E. G. Runyan, Washington D.C. . | 12.67      |  | 7.20              |  |
| Frank T. Shutt, Ottawa .. ..    | 12.30      |  | 6.97              |  |
| A. T. Charron, Ottawa .. ..     | 12.55      |  | 7.08              |  |
| R. W. Thatcher, Washington ..   | 13.02      |  | 7.96              |  |
|                                 | 13.07      |  | 8.01              |  |

#### Determination of Sulphur ( $SO_3$ ).

The results obtained for sulphur in the samples as sent out are given in the following table:—

TABLE IV.—Determination of Sulphur ( $SO_3$ ).

| Analyst.                                    | Corn bran.                  |                                    | Cotton-seed meal.           |                                    |
|---------------------------------------------|-----------------------------|------------------------------------|-----------------------------|------------------------------------|
|                                             | Pro-<br>visional<br>method. | Modified<br>provisional<br>method. | Pro-<br>visional<br>method. | Modified<br>provisional<br>method. |
|                                             | Per cent.                   | Per cent.                          | Per cent.                   | Per cent.                          |
| J. H. Gibboney, Virginia                    | 0.27                        | 0.32                               | 1.06                        | 1.07                               |
|                                             | 0.28                        | 0.34                               | 1.07                        | 1.10                               |
|                                             | 0.28                        | 0.30                               | 1.01                        | 1.00                               |
| E. G. Runyan, Wash-<br>ington, D.C. . . . . | 0.43                        | 0.33                               | 1.04                        | 1.08                               |
|                                             | 0.42                        | 0.34                               | 1.05                        | 1.08                               |
|                                             | 0.44                        | 0.32                               | 1.04                        | 1.14                               |
| Frank T. Shutt, Ottawa                      | 0.40                        | 0.15                               | 1.27                        | 1.10                               |
|                                             | 0.40                        | 0.13                               | —                           | 1.05                               |
| A. T. Charron, Ottawa                       | 0.34                        | 0.24                               | 1.24                        | 1.00                               |
|                                             | 0.34                        | 0.24                               | 1.34                        | 1.08                               |
| G. S. Fraps, North<br>Carolina .. . . .     | —                           | 0.41                               | —                           | 1.03                               |
|                                             | —                           | 0.40                               | —                           | 1.00                               |
|                                             | —                           | —                                  | —                           | 1.03                               |
|                                             | —                           | —                                  | —                           | 1.08                               |
| R. W. Thatcher, Wash-<br>ington . . . . .   | 0.27                        | 0.24                               | 0.99                        | 0.97                               |
|                                             | 0.23                        | 0.25                               | 1.00                        | 0.97                               |

#### Remarks of Analysts.

R. J. Davidson, Virginia.—We tried several determinations on these substances by Eschka's method, but found the result too low. On 2 grm. samples we got the following weights of barium sulphate: 0.0240, 0.216, and 0.0209 grm. just about one-third of what they should be.

R. W. Thatcher, Washington.—My experience with the two methods suggested for the determination of sulphur is that they give practically identical results, those obtained by the calcium acetate ignition method being very slightly lower. The use of calcium acetate allows much more rapid burning to whiteness, since the potassium nitrate present in the other method fuses very easily, and thus encloses particles of carbonaceous matter, which are finally removed only after a somewhat tedious process.

Frank T. Shutt, Central Experimental Farm, Ottawa, Canada.—We found great difficulty in obtaining a white ash by Method II. Even after prolonged ignition the ash remained grey, and on treatment with acid showed the presence of carbon. You will notice that invariably higher results were obtained by Method I. As far as one can judge from duplicate determinations and general conduct and appearance of the material during treatment, Method I. seems to be very satisfactory. To lessen the danger from loss while frothing (treatment with nitric acid on water-bath) a 3-inch porcelain evaporating dish was used. It will be found much safer than a 2½-inch dish. Ignition in a muffle furnace proved easier and quicker than with the blast-lamp.

The agreement between the figures obtained by different analysts by both methods is not satisfactory. The trouble is with the methods. They are not very accurate. As will be shown below, the results given by the nitric acid method are much lower than they should be.

(To be continued).

## PRECIPITATION AND SEPARATION BY WEAK ORGANIC BASES.\*

By E. T. ALLEN.

A LARGE number of organic bases are known, which may serve as quantitative precipitants of many metallic hydroxides, though they usually present no advantage over ammonia or the fixed alkalis. Of late, more especially (*Journ. Am. Chem. Soc.*, xxiv., 540, and xxi., 776), some of them have found use for analytical separations, where the inorganic reagents are not applicable. We have, between the organic and inorganic bases, a nearly complete analogy. The tetrammonium compounds are well known to be highly dissociated in aqueous solution, giving, in a marked degree, the characteristic hydroxyl reactions, like the hydroxides of the alkalis and thallium. A second weaker class of bases comparable, in general, with ammonia are the paraffin amines. These also are strongly alkaline in reaction, and capable of precipitating all but the strongest metallic bases, whose solubility products are too high to be reached by such concentrations of hydroxyl as these reagents afford.

A third class of bases in which the aromatic amines, quinoline, naphthylamine, phenylhydrazine, &c., may be named, have no soluble analogues in the inorganic field, though they are akin to the bases formed by the metals of the third and fourth groups of the periodic system.

With indicators they give either a faint alkaline reaction or none at all. When a metallic salt is precipitated by such a base, say aniline, the solution remains acid even when a considerable excess of the reagent has been added. It is plain, therefore, that only those bases which can be thrown down in acid solution, can be precipitated here. All chemists are familiar with the precipitation of certain hydroxides by the action of water alone. Such reactions

\* From the *Journal of the American Chemical Society*, xxv., No. 4.

are always reversible. We may instance the case of titanium sulphate,  $\text{Ti}(\text{SO}_4)_2 + 4\text{H}_2\text{O} = 2\text{H}_2\text{SO}_4 \cdot \text{Ti}(\text{OH})_4$ .\*

The complete precipitation of the titanium evidently depends on the reduction of the concentration of free acid to a point where the titanium hydroxide is no longer appreciably soluble. Let us suppose the precipitation to be incomplete. If now we should add to the system a weak base like aniline, a certain quantity of the free acid would be converted into neutral salt, and thus the concentration of the acid would be diminished, though an appreciable quantity would always remain unless a very great excess of reagent were used, for, according to the principles of hydrolysis, the salt of a weak base must be partially decomposed by water into free base and free acid. Thus,  $\text{C}_6\text{H}_5\text{NH}_3\text{Cl} + \text{C}_6\text{H}_5\text{NH}_2 + \text{HCl}$ .

The precipitation of titanium will therefore go further, in fact, to completion when a proper quantity of aniline has been added, because in small concentrations of acid, titanium hydroxide is practically insoluble. The degree of the hydrolysis of the aniline hydrochloride is conditioned by temperature, by the degree of dilution, and by excess of free base. Speaking in general of this class of weak bases, it is, of course, true that the degree of hydrolysis, in other words the concentration of free acid in solutions of their salts, depends on the strength of the base. Some organic bases are so weak that they would doubtless be of no service for any quantitative precipitation, but those whose hydrochlorides are hydrolysed to an extent of several per cent in dilute solutions form a group well adapted for the separation of weak from strong metallic bases. For the strong bases like magnesia, the alkaline earths, &c., are incapable of existence with free acid, while the weak ones, whose salts are more or less hydrolysed in aqueous solution, are practically insoluble in such concentrations of acid as one can readily obtain by the above means. This is the principle of most analytical separations by weak organic bases, and the most important one involved in the experimental part of this paper. Of course there are cases in which the precipitate by an organic base re-dissolves in excess. Thus several paraffin amines re-dissolve the hydroxides of aluminium, copper, silver, zinc, &c., and it is possible some of the weaker bases may yield separations on this principle, though no cases of the kind are known to me.†

In the study of the separations in the sequel, it seemed of interest, not merely to determine the necessary empirical conditions, but also to inquire how much acid is freed from the salts of certain weak bases—the precipitants—under definite conditions of dilution, temperature, and excess of the base. Thus the hydrolytic constant,—

$$K = \frac{\text{BOH} \times \text{HCl}}{\text{V}(\text{BCl})}$$

of aniline and of phenylhydrazine hydrochloride was determined in N/10 solutions at a temperature of 40° C. In this way could be made an approximation to the concentration in free acid possessed by the solutions in which the separations were made—concentrations in which certain weak metallic bases remain without dissolving appreciably.

#### Separation of Aluminium from Iron.

Several years ago Campbell and Hess (*Journ. Am. Chem. Soc.*, xxi., 776) described a method for precipitating aluminium in the presence of iron after the latter had been reduced to the ferrous state by sulphurous acid. The reagent used was phenylhydrazine. The results appeared satisfactory, and, as a good method for the direct determination of aluminium in the presence of iron was needed, Dr. W. F. Hillebrand suggested that I study this separation more closely, especially in regard to its application to rock analysis, where titanium is nearly always present, and sometimes zirconium. I desire in this place to express to Dr. Hillebrand my best thanks for this suggestion.

\* The principle involved is the same whether the precipitate be a hydroxide or a basic salt.

† In her statement that phenylhydrazine throws down, from thorium nitrate, a "canary-yellow precipitate, easily soluble in excess," Miss Jefferson appears to be in error (*Journ. Am. Chem. Soc.*, xxiv., 546).

In brief, my results indicate that it is practically impossible to separate the iron completely in one precipitation. With a double precipitation the results are good. When the quantity of precipitated alumina is large, there is apt to be a loss, which I ascribe to the long contact with the faintly acid wash solution which is necessary to remove the iron. Thus I have obtained on certain rocks results which were considerably lower than those carefully determined by difference in the usual way. On the other hand, from mixtures of iron, aluminium, titanium, and zirconium salts made up from standard solutions the results have been satisfactory. This discrepancy has not thus far been explained. The method is excellently adapted to the separation of very small quantities of aluminium, such as a milligram, or even less, from a large excess of iron, a point of considerable practical importance.

In the following tests, standard solutions of ferric and aluminium chlorides were used. The former was prepared from piano wire by dissolving in pure hydrochloric acid, filtering, oxidising with chlorine, and driving out the excess from the hot solution by a current of air. No alumina or other bases of similar strength could be detected in this solution. It was standardised by precipitation with ammonia. The aluminium chloride was prepared by dissolving "C. P." metal in hydrochloric acid, filtering off the silicon, and diluting. The exact strength was determined by precipitation with ammonia. The solution contained only a trace of iron.

To the conditions laid down by Campbell and Hess a few additions might be made. The volume of the solution may vary, according to the quantity of alumina to be precipitated, from 100 to 200 c.c. It should be heated and reduced by adding saturated ammonium bisulphite. From 5 to 20 drops, according to the quantity of iron, may be used. If the solution turns deep red (ferric sulphite) it is not acid enough, and a few drops of hydrochloric acid should be added, for the sulphite itself does not reduce ferric salts, at least not with rapidity. Now quickly bring to neutrality with ammonia, and then add several drops of dilute hydrochloric acid. If this last operation is done too slowly, the oxygen of the air helps to form a little ferric hydroxide which does not always readily dissolve in the dilute acid. Finally, add from 1 to 3 c.c. phenylhydrazine,\* according to the weight of the alumina to be precipitated. If too little has been used, a few drops added to the filtrate will disclose the mistake. Stir until the precipitate has become sufficiently flaky, and allow to settle. The supernatant liquid will now be plainly acid to litmus. One need not be disturbed if the precipitate has a brownish colour, for it is not due to ferric hydroxide, but to the colouring matter contained by all phenylhydrazine which has not been freshly distilled. When the determinations are allowed to stand too long, the air increases this oxidation product, and a brown insoluble scum forms on the surface of the liquid and on the sides of the vessel, which is rather troublesome to the analyst. Fortunately equilibrium appears to be established in a short time. The vessels need not stand more than an hour at any rate.†

#### Precipitation of Alumina alone by Phenylhydrazine.

1 c.c.  $\text{AlCl}_3 = 0.005001$  grm.  $\text{Al}_2\text{O}_3$ .

| Taken.                                          | Found. | Error.   |
|-------------------------------------------------|--------|----------|
|                                                 | Grm.   | Grm.     |
| 1 50 c.c. = 0.2500 grm. $\text{Al}_2\text{O}_3$ | 0.2487 | - 0.0013 |
| 25 c.c. = 0.1250 grm. $\text{Al}_2\text{O}_3$   | 0.1236 | - 0.0014 |
| 5 c.c. = 0.0250 grm. $\text{Al}_2\text{O}_3$    | 0.0254 | + 0.0004 |

The tendency toward low results is here plainly visible.

(To be continued).

\* The reagent should, of course, be free from inorganic impurities which could disturb the results. The author found one sample, which, after persistently giving high results, was proved to contain tin, which had probably been used in its preparation.

† The precipitate is washed by a solution of phenylhydrazine sulphite, made by adding cold saturated sulphurous acid to a little phenylhydrazine until the crystalline sulphite first formed dissolves in the excess. The solution has an acid reaction. Five to 10 c.c. of this are used in 100 c.c. hot water. See Campbell and Hess, *loc. cit.*

# THE ANALYSIS OF COMMERCIAL NICKEL.

By A. HOLLARD.

**Impurities.**—Copper, cobalt, iron, alumina, lime, magnesia, sulphur, silica, carbon, arsenic, antimony.

**Estimation of the Nickel.**—5 grms. of the metal are placed in a Bohemian beaker 65 m.m. diameter and 180 m.m. in height. It is in this same glass that the attack of the metal, the evaporation, and its subsequent electrolysis will be successively carried out. The great height of the glass is for the purpose of stopping all loss by projections during the electrolysis. Thanks to these precautions there is not the slightest loss of metal.

We pour a mixture of 25 c.c. of nitric acid and 25 c.c. of water over the nickel, and cover the beaker immediately with a glass funnel, of which the edges rest on the interior of the beaker, and thus form a little gutter in which a few drops of water make a perfect hydraulic joint. Heat gently during the whole of the attack, and at the end raise the temperature to boiling. When the attack is finished withdraw the flame and leave the funnel in the glass for at least a quarter of an hour longer, so that the condensing vapours will wash or rinse the underside of the funnel. Then add 10 c.c. of  $\text{H}_2\text{SO}_4$  and evaporate over a small flame until white sulphuric fumes are given off abundantly. There still remains a large excess of  $\text{H}_2\text{SO}_4$ . Let cool and take up with water, then add ammonia in excess by 2 or 3 c.c. Boil for an instant to precipitate the iron completely, after having replaced the funnel in the beaker so as to stop projections. Then add 25 c.c. of ammonia at  $22^\circ$ , then a few c.c. of pure peroxide of hydrogen, which prevents the deposition of carbon with the nickel; dilute to 300 c.c., then electrolyse hot (at about  $90^\circ$ ) with a current of 1 ampère with spiral and cylindrical electrodes; the cathode is made of platinum gauze dulled in the sand blast. After a few hours the nickel is all deposited. The rapidity with which the deposit is made is on account of the shape of our electrodes, and also because the metallic gauze allows a free circulation of the gases and the liquid.

The precipitate of silica and of the hydrates of iron and alumina present in the liquid does not interfere with the deposition of the nickel; on the contrary, the current frees this precipitate from the entangled nickel much more completely than would be done by filtration and repeated washings. Once the nickel is deposited, the precipitate is filtered and re-dissolved in the smallest possible quantity of sulphuric acid, neutralise with ammonia, which must be added in excess, and add the whole to the original solution. Electrolyse as before, using the same cathode already covered with almost the whole of the nickel. A test with sulphhydrate of ammonium will indicate the end of the electrolysis. While still leaving the electrodes hanging from their support, the beaker containing the electrolytic bath is withdrawn and replaced with a beaker filled with distilled water; the current must continue to pass for about five minutes, and this frees the gauze from all salts it might retain. The cathode is then plunged for a few minutes into a second beaker of distilled water, then into absolute alcohol; dry and weigh. In this manner we get the whole of the nickel and cobalt present in the original metal, as well as the copper. So the weight of the copper and the cobalt must be subtracted after being estimated, as will be described further on. As for the arsenic and the antimony, they remain with the hydrate of iron in the form of insoluble arseniate and antimoniate of iron.

**Estimation of the Copper.**—The electrolytic deposit just obtained is dissolved in a mixture of 50 c.c. of nitric acid at  $36^\circ$  and 50 c.c. of water. Electrolyse with a current of 1 ampère, after having diluted to 300 c.c. Weigh the deposited copper after having washed and dried it as described above.

**Estimation of the Cobalt.**—The nitric solution freed from copper is evaporated to a syrupy consistence on a sand bath. Take up with water, and make alkaline with potash, then dissolve in acetic acid; finally add 150 to 200 c.c. of a 50 per cent solution of nitrite of potash. After standing

all night the precipitation is complete; filter, wash with a solution of acetate and nitrite of potassium, then dissolve in a mixture of 1 volume of  $\text{H}_2\text{SO}_4$  and 1 volume of water, and 3 to 4 volumes of hydrochloric acid. Evaporate until the appearance of abundant fumes of sulphuric acid. The sulphate of cobalt is then electrolysed under the same conditions as the nickel, already described.

**Estimation of the Iron.**—In the mother-liquors, freed from nickel, cobalt, and copper, there is still the precipitate of silica, alumina, and iron, as well as the arsenic and antimony. This precipitate is filtered, washed, and dissolved in dilute sulphuric acid. The antimony and the arsenic are precipitated by sulphuretted hydrogen, filtered and washed, then the sulphuretted hydrogen is driven off from the filtrate by heating. The liquid is treated with 5 grms. of citric acid, from 25 to 50 c.c. of a saturated solution of sulphurous acid, and 25 c.c. of ammonia at  $22^\circ$ ; then dilute sulphuric acid is added drop by drop until the solution is neutral. Make alkaline with a few cubic centimetres of ammonia, dilute up to 300 c.c., and electrolyse at a temperature of about  $40^\circ$  with a current of 1 ampère. After a short time the iron is deposited integrally. As it carries with it a little carbon and also a little platinum from the dissolution of the anode under the influence of the sulphurous acid, it cannot be weighed; it is therefore estimated volumetrically; for this purpose it is dissolved in dilute sulphuric acid, boiled and filtered (the filter retains the platinum and the carbon deposited with the iron, which would otherwise act on the  $\text{KMnO}_4$ ), and left to cool in a current of  $\text{CO}_2$  and in the presence of a small piece of zinc, sufficient to reduce the iron. When the whole of the zinc is dissolved and the solution is cold, it is titrated with permanganate of potash.

**Estimation of the Alumina.**—The mother-liquors from the electrolysis of the iron are evaporated to dryness with an excess of sulphuric acid. To destroy the remainder of the organic matter the residue is taken up with a mixture of equal volumes of sulphuric and nitric acids, then again evaporated to dryness. The residue is dissolved in water acidulated with hydrochloric acid, and the filtered solution is treated with hydrochlorate of ammonia and ammonia, then boiled. The alumina is precipitated, it is washed, filtered, and weighed in the state of  $\text{Al}_2\text{O}_3$ .

**Estimation of the Lime.**—The mother-liquors which have been freed successively from the nickel, cobalt, iron, and aluminium, still contain the whole of the lime. These liquors are concentrated, but not sufficiently to cause crystallisation of the salts—then precipitated with oxalate of ammonia. The well-washed oxalate of lime is placed, with its filter, in a conical flask, and sulphuric acid at one-fifth strength (by volume) is added. The oxalic acid is titrated with permanganate of potash at  $35^\circ$  to  $40^\circ$ , after having added a little manganous sulphate to the flask to accelerate the action of the permanganate.

**Estimation of the Magnesia.**—The mother-liquors, free from lime, are treated with arseniate of soda, which precipitates the magnesia in the form of ammonio-magnesian arseniate. Filter, wash, and dissolve the precipitate in 150 c.c. of hydrochloric acid at  $22^\circ$ ; this solution is then distilled for arsenic in the presence of 15 grms. of ferrous sulphate (see Hollard and Bertiaux, *Bull. Soc. Chim.*, 1900, vol. xxiii., p. 300).

From the arsenic, estimated volumetrically, we calculate the magnesia,  $\text{MgO} = \text{As} \times 0.538$ .

**Estimation of the Sulphur and the Silica.**—Five to 10 grms. of the metal are attacked with nitric acid diluted with its own volume of water. Evaporate on the water-bath after having added about 25 c.c. of hydrochloric acid, and drive off the  $\text{HNO}_3$  by repeated evaporations with hydrochloric acid. The residue is treated with 2 to 3 c.c. of hydrochloric acid, then with water. Filter to remove the silica and wash with warm water.

The filtrate contains only a portion of the sulphur, the other part being retained by the silica. The filter containing the silica is dried, then burnt with its contents, after impregnating them with a solution of nitrate of ammonia,

which prevents the formation of volatile sulphurous acid. The whole is fused with 2 grms. of carbonate of soda. After complete fusion, allow to cool, and treat the melted mass with water and hydrochloric acid in excess, evaporate to dryness on the water-bath, take up with 2 or 3 c.c. of hydrochloric acid, then with water, filter the silica and wash it with water. The filtrate which contains the sulphur present in the silica is added to the first filtrate, and the mixture is precipitated with chloride of barium at boiling temperature; allow to stand for a night in a warm place. Filter and weigh the sulphate of baryta.

As for the silica, it is dried, calcined, and weighed. It is then treated with hydrofluoric acid and a few drops of sulphuric acid. Evaporate, calcine, and weigh. From the loss of weight we calculate the amount of silica.

*Estimation of the Carbon.*—Proceed as for the estimation of the carbon in cast-iron or steel.

*Estimation of the Arsenic.*—Distil the arsenic by heating the nickel with ferric sulphate and hydrochloric acid. (see Hollard and Bertiaux, *Bull. Soc. Chim.*, 1900, vol. xxiii., p. 300).

*Estimation of the Antimony.*—The liquid freed from arsenic by distillation, and which contains all the antimony, is treated with chloride of zinc, then distilled in a current of hydrochloric acid gas (see Hollard, *Ann de Chim. Analytique*, 1900, p. 330). The chloride of antimony distilled over is precipitated with sulphuretted hydrogen, and then estimated electrolytically (see *Bull. Soc. Chim.*, 1903, vol. xxix., p. 262).—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 22.

## NOTICES OF BOOKS

*La Grande Industrie Chimique Minérale.* ("The Industries of Mineral Chemistry"). By E. SOREL. Paris: C. Naud. 1904.

The subjects of which this volume treats include potassium, sodium, and the halogen elements and their compounds. The first chapters describe the properties and methods of extraction of sea salt and rock salt, and the preparation of potashes, iodine, and bromine. The preparation of sulphate of sodium is then considered, and the manufacture of hydrochloric acid. Considerable space is devoted to the description of the Leblanc soda process, which appears ill-advised when it is remembered that it is rapidly giving way to more recent and economical methods, though it may be some time before it is entirely superseded. The last part of the book deals with the manufacture of chlorine and the various recovery processes connected therewith. The information seems to be well up to date, and no doubt this volume will be found as useful as the preceding one by the same author, dealing with sulphur, nitrogen, and phosphorus, which appeared some two years ago.

*Alfred Werner's Theorie des Kohlenstoffatoms und die Stereochemie der Carbocyclischen Verbindungen.* ("Alfred Werner's Theory of the Carbon Atom, and the Stereo-chemistry of the Carbocyclic Compounds"). By Dr. ERNST BLOCK. Wien und Leipzig: Carl Fromme. 1903.

THIS short sketch of Werner's theory relating to the stereo-chemistry of the carbon compounds contains a very readable review and critical examination of the various theories of the structure of the benzene nucleus which have been put forward since the time of Kekulé. Regarding Werner's theory as the most satisfactory from every point of view, the author discusses it in detail, and upon the three formulæ suggested by Werner, Vaubel, and Sachs respectively, founds a new benzene formula which appears to combine the advantages of all three. In some cases, Dr. Block shows a tendency to write as if the last word had been written on the subject, but the original sugges-

tions contained in the book are certainly worth noting. The constitutions of naphthalene, anthracene, phenanthrene, and pyrene are also shortly discussed in the light of the new benzene theory.

*Hypochlorite und Elektrische Bleiche.* ("Hypochlorites and Electrical Bleaching"). By VIKTOR ENGELHARDT. Halle-a-S.: Wilhelm Knapp. 1903.

THE author of this monograph has divided his subject into two parts, to be treated in separate volumes. Keeping in mind the very different requirements of the technical electro-chemist and the manufacturer, he has reserved for the second part all details regarding practical applications and the methods of examination chemically of the raw materials, while this volume is devoted to the consideration of the technical and constructive aspect of the subject. A great number of specifications of patents granted in most European countries, and in America, are abstracted, and details of apparatus described at some length with copious illustrations; a chronologically arranged tabular statement of the main features of the most important patents brings the subject down to 1902. The specifications have been grouped under different headings in such a manner as to simplify greatly the study of the subject, and the book is a model of conciseness and convenience.

*Proceedings of the Chemical and Metallurgical Society of South Africa.* With Appendix. February, 1897—September, 1899. Vol. II. Johannesburg: Published by the Society. Edinburgh: R. W. Hunter. New York: *The Engineering and Mining Journal*. Pp. 928.

THE belated appearance of this volume, which was published only in October last, is explained by the Secretary to be due to the late war. The volume, he says, would have been published earlier but for the fact that the "copy" was left in Johannesburg during the war, and that a large number of the members being on active service in the various irregular regiments during that period, it was impossible for some of the papers to be revised until their return.

The volume begins with a list of officers and council for 1897-98-99-1902, and a list of members and associates in September, 1899. Then follow the records of the monthly meetings from February 20th, 1897, to September 16th, 1899, the papers read at these meetings, and the discussions thereon. These meetings are of very great value to the mining industry, for it is here that the different managers and chemists interchange ideas and give each other, and the world at large, the benefit of their experiences.

We feel safe in asserting that never has a goldfield been conducted on such scientific lines as the Witwatersrand. The old rule-of-thumb methods have had their day, and a good one it was; but now that science has come to the assistance of blind experience, gold is being won from tailings, slimes, and even tail-waters, to an extent that would fairly astonish the old-time mining captains and prospectors.

These results have been brought about to a very large extent by the "Chemical and Metallurgical Society of South Africa," and we heartily congratulate the society on having got through the trouble and stress of a prolonged period of fighting, though with somewhat impaired ranks, and feel confident that their labours will be as progressive in the future as they were valuable in the past.

*The Latin Grammar of Pharmacy.* By JOSEPH INCE, F.C.S., F.L.S., F.G.S. Tenth thousand, Eighth Edition. London: Baillière, Tindall, and Cox. 1903. Pp. 371.

We can imagine this volume being of use to medical and pharmaceutical students who have gone through their early school days without learning very much Latin, but to the average student who has had the usual liberal education, a



large part of this volume would be entirely superfluous. On the other hand, we doubt if a student of eighteen or nineteen years of age, who had not already learned Latin, would be able to do much good at so belated a period.

The first portion of the volume is simply an ordinary Latin grammar, but beginning with page 93 we come to the technical portion, "Essay on the reading of Latin prescriptions," followed by another on "Medical directions."

One of the most useful portions of the book is that containing the "reference vocabularies." This is in two parts, "Latin-English" and "English-Latin," and covers about 60 pages.

The book is conveniently arranged, and as a reference book it will no doubt be serviceable and worth a place on the book-shelf.

*A Short Manual of Analytical Chemistry* (Qualitative and Quantitative—Inorganic and Organic). By JOHN MURTER, Ph.D., F.R.S.E., F.I.C., &c. Ninth Edition, Illustrated. London: Baillière, Tindall, and Cox. 1903. Pp. 235.

THIS book, originally written for pharmaceutical students, has been so extended and improved as to rank as one of the most useful reference books extant for the general analytical practitioner. It is essentially not a book for beginners, and Chapter I. might be easily omitted as unnecessary. No extension has been made in this edition, but some alterations and improvements have been introduced as the result of experience. Advantage has also been taken of the use of certain new reagents (such as sodium peroxide) in simplifying manipulations.

## CORRESPONDENCE.

### WHAT MAY RESULT FROM THE STUDY OF RADIUM.

*To the Editor of the Chemical News.*

SIR,—Apropos of the interest evinced both by scientists and the public generally, as evidenced by their muster to listen to Sir Oliver Lodge recently, may I have the privilege of space to draw attention to an analogical position which existed about a century ago, when in 1815-1816, Dr. Prout propounded his famous and astounding theory, causing not less sensation than the historical prophecy of Sir William Crookes last summer at Berlin, who in concluding expressed the opinion that the atomic dissociation may again reduce matter to the protyle, the formless mist; and when speaking on the miracle of radium, as Mr. Boys, President of the Physical Section of the British Association, termed it in his address last September, called attention to the analogy between the electrons from radium and those emitted by comets, popularly called its "tail."

Dr. Prout's celebrated theory put in a nutshell was that the atoms of the various elements were the essence in more or less concentration of one primordial substance, which he took for argument as hydrogen. Thus, according to atomic weight, an oxygen atom would consist of 16 hydrogen atoms compounded, whilst 32 atoms of hydrogen would be required to produce one of sulphur. By supposing all these atoms to be of one uniform size, as inferred from Avogadro's law—"that equal volumes of gases contain the same number of molecules"—we had simply in the various elements physical compounds made up of atoms of uniform size, but of greatly varying potential. It was subsequently argued, particularly by Stas, who made some careful researches, that Prout's theory was unsound, or else the atomic weights of the various elements would be exact whole numbers, whereas there were fractions to be accounted for. Again, Dumas, in 1878, reverted to the "primordial atomic" theory, and brought arguments by which several of Stas's results were brought more

closely into line with Prout's theory. Others of no small repute as chemists and physicists have argued for and against the theory, which although, as far as I know, never satisfactorily refuted, has been allowed to lie dormant.

Now if it is established beyond argument, as Sir Oliver tells us, that atoms are composed of electrons, of which a thousand million million packed closely would be required to make an atom in bulk, and moreover from the experiments of Prof. Rutherford last February, it might be inferred that the Alpha rays of radium became another element (helium also of recent discovery), because the atomic weight of the electrons of those rays was twice that of hydrogen; which inference has now been set beyond doubt by Sir W. Ramsay and Prof. Soddy, having watched the spectrum of helium gradually develop in an excited vacuum tube into which only radium emanation had been placed. Further remarkable evidence of this change of state results from the observations by Sir Wm. and Lady Huggins, of the spectrum of radium in air, giving the spectrum line by line of nitrogen. And, as Prof. E. Rutherford says, the energy displayed by radium emanation must be internally stored up. Is not all this evidence marvellously analogous to the crude theory of Prout? And do not the researches of Prof. Rutherford and other leading scientists point to the time when, if not already present, the celebrated theory of our philosopher of nigh a century ago is on the very doorsteps of truth, and all atoms are made up of a simple substance, but of varying potential, even as the potential of radium is above that of any known substance, and is naturally falling, very, very, almost inestimably, slowly through the other states, each state of which constitutes another element?—I am, &c.,

H. BARKER LAKE.

77, Colmore Row, Birmingham.  
January 14, 1904.

### WHY ARE SOME ELEMENTS SO MUCH MORE ABUNDANT THAN OTHERS?

*To the Editor of the Chemical News.*

SIR,—It is well known that different elements occur in very different quantities. Some elements are excessively rare, others are excessively abundant. Now what is the reason of this? Why are not all elements equally abundant? Is not perhaps a reason that some elements are more stable than others?

So that the most stable elements occur in the greatest quantity, the most unstable in minute quantities because, of the more rapid transformation of the unstable elements into other forms of matter?

A perusal of the researches of Sir William Crookes, Sir William Ramsay, and others certainly inspire such thoughts.

Indeed, the phenomena may be part of that universal decay of elements so grandly pictured by Sir William Crookes—the elements decaying at different rates, just as different substances evaporate at different rates. The bearing of such a conception upon geology and the past history of the earth is obvious. A slow continuous process acting for many ages can produce the mightiest effects.—I am, &c.,

GEOFFREY MARTIN.

Institute of Chemistry of Great Britain and Ireland.—Final (A.I.C.) Examination, April, 1904.—A Final Examination, in all branches except that of Biological Chemistry, will be held at the Laboratories of the Institute, commencing on Tuesday, the 12th day of April, 1904, provided that a sufficient number of Candidates enter their names for the same. The Examination will be open only to Candidates who have complied with the Regulations. Applications should be forwarded not later than Tuesday, the 23rd day of February, 1904. For further particulars apply to the Registrar, Institute of Chemistry, 30, Bloomsbury Square, London, W.C.

## MEETINGS FOR THE WEEK.

MONDAY, 25th.—Society of Arts, 8. (Cantor Lectures), "Oils and Fats—their Uses and Applications," by J. Lowkowitzsch, Ph.D., &c.  
 TUESDAY, 26th.—Royal Institution, 5. "Development of Animals," by Prof. L. C. Miall, F.R.S.  
 WEDNESDAY, 27th.—Society of Arts, 8. "Ice-breakers and their Services," by Arthur Gulston.  
 THURSDAY, 28th.—Royal Institution, 5. "The Flora of the Ocean," by G. R. M. Murray, F.R.S.  
 FRIDAY, 29th.—Royal Institution, 9. "The Marshes of the Nile Delta," by David George Hogarth, M.A.  
 SATURDAY, 30th.—Royal Institution, 3. "British Folk Song" (with Vocal Illustrations), by J. A. Fuller Maitland, M.A.

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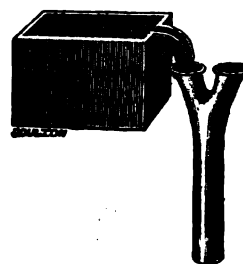
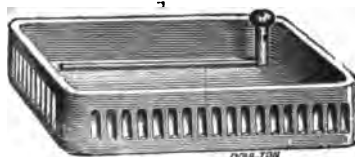
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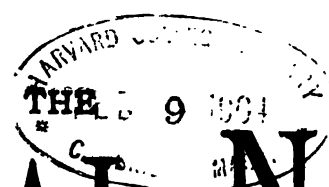
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### CONTENTS.

| ARTICLES:—                                                                                                                                         | PAGE |
|----------------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Causes of the Analogy between Fluorine and Oxygen, by Geoffrey Martin, B.Sc. (Lond.)                                                           | 49   |
| Use of Bismuth as a means of Separation in the Rare Earth Series, by G. Urbain and H. Lacombe                                                      | 52   |
| London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.                                                                       | 52   |
| Report on Ash, by G. S. Fraps                                                                                                                      | 54   |
| A New Method for the Attack of Galenas and Chalcopyrites, by C. Boucher                                                                            | 56   |
| PROCEEDINGS OF SOCIETIES—                                                                                                                          |      |
| PHYSICAL SOCIETY.—Notes on Non-homocentric Pencils and the Shadows produced by them, an Elementary Treatment of the Standard Astigmatic Pencil—&c. | 57   |
| CORRESPONDENCE.—Why are some Elements so much more Abundant than others?                                                                           | 58   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                              | 58   |
| MISCELLANEOUS                                                                                                                                      | 59   |
| MEETINGS FOR THE WEEK                                                                                                                              | 60   |

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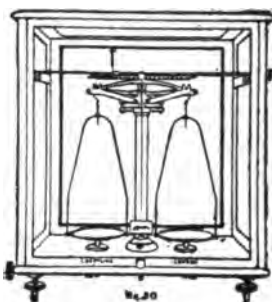
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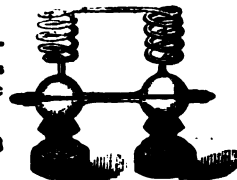
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VOL. LXXXIX., No. 2305.

## THE CAUSES OF THE ANALOGY BETWEEN FLUORINE AND OXYGEN.

By GEOFFREY MARTIN, B.Sc. (Lond.).

MR. LENHER, in the *CHEMICAL NEWS* for December 24th, 1903, vol. lxxxviii., p. 307, showed that, whereas gold gives at ordinary temperatures a fairly stable chloride, the corresponding fluoride is very unstable, and he expressed some surprise that this should be so. The law underlying this observation is given below. Moissan some years ago (*Bull. Soc. Chim.*, 1891, [3], v., 880) pointed out that, of all the halogens, fluorine is the one which resembles oxygen most closely in its general properties and in its compounds.

Being curious to know what it is that causes fluorine to resemble oxygen more closely than chlorine or bromine does, I proposed to myself the following questions:—

A. Does not fluorine resemble oxygen more closely than chlorine resembles oxygen, because the fluorine approaches the oxygen more closely than the chlorine does, as regards the intensity of the force with which it attracts a given radicle? In other words, does not the chemical similarity of two elements arise from the fact that they both attract the same radicles with nearly the same intensity of force?

B. Seeing that chemically similar elements and compounds, as a rule, have about the same degree of volatility and fusibility, is not this because there is a connection between the intensity of the chemical forces which an atom is capable of exerting and the degree of volatility of the compounds into which it enters? And in particular, would not this connection exhibit itself among the fluorides, chlorides, and oxides, in such a manner that those compounds which approach each other nearest in the intensity of the force with which the atoms in the molecules are attracted together, be those which approach each other nearest as regards fusibility and volatility?

That is to say, is it not the internal atomic forces in the molecule which determine the external forces with which the molecules themselves are attracted together?

The answer to both questions is in the affirmative, as will be seen from the following data:—

A. First we will show that fluorine and oxygen approach each other more closely in the intensity of the force with which they attract the same radicles than do chlorine and oxygen.

This may be established by three lines of argument:—

- That founded on a comparison of the heats of formation of corresponding fluorides, chlorides, and oxides.
- That founded on the fact that O and F both tend to make an element with which they combine exhibit a higher grade of valence than Cl can make it exhibit.
- That founded on the chemical stability of corresponding fluorides, chlorides, bromides.

We will consider each briefly.

a. The heats of formation of the fluorides (so far as they are known) stand closer to the heats of formation of the corresponding oxides than to those of the corresponding chlorides. Thus, arguing that the intensity of force which holds the atoms together in the fluorides stands nearer to that which holds the atoms together in the corresponding oxides than in the corresponding chlorides.

For example, we have:—

(H, Cl) = 22; (H, F) = 37·6; (H, O) = 34·2.

b. Oxygen and fluorine both resemble each other in that

they tend to make the elements with which they combine exhibit a higher grade of valence than Cl can make them exhibit.

For example, the stablest oxide of Mn at ordinary temperatures is  $Mn_2O_3$ . Also, the stable fluoride is  $MnF_3$ , whereas the stablest chloride is  $MnCl_2$ ,  $MnCl_3$  being an excessively unstable body spontaneously decomposing at ordinary temperatures to  $MnCl_2$  and Cl. Again,  $SO_3$  is at ordinary temperatures a quite stable body, not parting with O below a red heat. Also  $SF_6$  is a very stable body. Whereas  $SCl_6$  is too unstable to exist at ordinary temperatures, and  $SCl_4$  is only capable of existing below  $-20^\circ C$ . Similarly,  $P_2O_5$  is at ordinary temperatures by far the stablest oxide of P; so also  $PF_5$  is the stablest fluoride of P; on the other hand,  $PCl_5$  is easily decomposed to  $PCl_3$ .

The stablest oxide of I is  $I_2O_5$ , and the stablest fluoride is  $IF_5$ . But  $ICl_5$  is so unstable that it seems incapable of existing at ordinary temperatures.  $ICl_3$  is also unstable, easily decomposing when heated. Only  $ICl$  seems to be stable enough to bear heating. (Notice that the fluorides follow the oxides and not the chlorides as regards stability).

These facts can be best interpreted to mean that both O and F exert nearly the same intensity of force on an element, so that when O is capable of making the element give rise to a stable compound of a high valency grade, F is also capable of doing the same thing.

Whereas the intensity of the force exerted by Cl on the same element is much less, so that the corresponding high valency compounds of Cl are much less stable than those of O and F.

c. The chemical stability of compounds depends upon the intensity of the forces with which the atoms are attracted together in the molecule. We will show that when we contrast an oxide and the corresponding fluoride, on the one hand, with the same oxide and the corresponding chloride on the other, the oxide and fluoride approach each other much more closely as regards their stability than do the oxide and chloride. In fact, when the oxides of an element are unstable and the chlorides stable, then the fluorides follow oxides in being unstable. And conversely, when the oxides are stable and the chlorides are unstable, the fluorides follow the oxides in being stable.

So that O and F approach each other more closely than do O and Cl in the intensity of the force with which they attract the same radicles. We will now illustrate this.

1. The following examples show that when the oxides are less stable than the chlorides, the fluorides are also less stable than the chlorides:—

$Na_2O$ ,  $NaCl$ ,  $NaF$ .—The fact that the heat evolved when Na combines with an equivalent of O is much less than the heat evolved when Na combines with an equivalent of Cl, [(Na, O) = 49·8, (Na, Cl) = 97·7], shows that Cl is attracted to the Na with a greater force than is O. The fact that NaCl is hardly acted on at all by superheated steam, whereas NaF is partially decomposed, indicates that in NaF the F is attracted to the Na with a force less than that with which the Cl is in NaCl.

$K_2O$ ,  $KCl$ ,  $KF$ .—We have (K, O) = 47·45, (K, Cl) = 105·6, so that KCl is very much more stable than  $K_2O$ . Also KCl is very much more stable than KF; for water at ordinary temperatures is capable of decomposing KF, whereas KCl is only decomposed by water at a red heat.

$Ag_2O$ ,  $AgCl$ ,  $AgF$ .—We have (Ag, Cl) = 29·00, (Ag, O) = 2·950, which indicates that AgCl is very much more stable than  $Ag_2O$ . Again,  $Ag_2O$  is completely decomposed at  $290^\circ$ , whereas AgCl is not decomposed at  $1735^\circ$  (white heat).

Similarly, AgF is like  $Ag_2O$ , much less stable than AgCl, being completely decomposed to Ag when heated in the air.

It would appear from Mr. Lenher's results (*vide supra*) that  $AuF_3$  in this respect resembles AgF, but is even more unstable than this body, because  $Au_2O_3$  is more unstable than  $Ag_2O$ . See also below,  $HgF_2$ .

TABLE I.—Showing that Non-metallic Fluorides are more Volatile than Non-metallic Chlorides.

| Compound.                             | Physical state. | Volatility.               |
|---------------------------------------|-----------------|---------------------------|
| PF <sub>3</sub> .. ..                 | Gas             | —                         |
| PCl <sub>3</sub> .. ..                | Liquid          | Boils at 78°              |
| PF <sub>5</sub> .. ..                 | Gas             | —                         |
| PCl <sub>5</sub> .. ..                | Solid           | Sublimes at about 147°    |
| POF <sub>3</sub> .. ..                | Gas             | —                         |
| POCl <sub>3</sub> .. ..               | Liquid          | Boils at 107.5°           |
| SiF <sub>4</sub> .. ..                | Gas             | Boils at -107°            |
| SiCl <sub>4</sub> .. ..               | Liquid          | Boils at 59°              |
| BF <sub>3</sub> .. ..                 | Gas             | —                         |
| BCl <sub>3</sub> .. ..                | Liquid          | 18.5°                     |
| CF <sub>4</sub> .. ..                 | Gas             | —                         |
| CCl <sub>4</sub> .. ..                | Liquid          | 78°                       |
| SF <sub>6</sub> .. ..                 | Gas             | Boils a little above -55° |
| SCl <sub>2</sub> .. ..                | Liquid          | Boils at +139°            |
| SOF <sub>2</sub> .. ..                | Gas             | " -32°                    |
| SOCl <sub>2</sub> .. ..               | Liquid          | " +78.8°                  |
| SO <sub>2</sub> F <sub>2</sub> .. ..  | Gas             | " -52°                    |
| SO <sub>2</sub> Cl <sub>2</sub> .. .. | Liquid          | " +82°                    |
| AsF <sub>3</sub> .. ..                | Liquid          | " 63°                     |
| AsCl <sub>3</sub> .. ..               | Liquid          | " 130°                    |

TABLE II.—Showing that Non-metallic Oxides are usually more Volatile than the corresponding Chlorides.

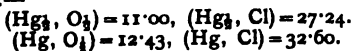
(Data is largely taken from HENRY, *Phil. Mag.*, 1885, [5], xx., 81).

| Compound.                              | Physical state.                            | Volatility.   |
|----------------------------------------|--------------------------------------------|---------------|
| OO .. ..                               | Perfect gas, formerly considered permanent | B.p. -183° C. |
| OCl <sub>2</sub> .. ..                 | Gas, easily liquefied                      | " -17°        |
| SO <sub>2</sub> .. ..                  | Gas                                        | " -10°        |
| SOCl <sub>2</sub> .. ..                | Liquid                                     | " +82°        |
| SO <sub>3</sub> .. ..                  | Solid                                      | " 46°         |
| SO <sub>2</sub> Cl <sub>2</sub> .. ..  | Liquid                                     | " 82°         |
| CO <sub>2</sub> .. ..                  | Gas                                        | Boils -78°    |
| COCl <sub>2</sub> .. ..                | Liquid                                     | " 8°          |
| CCl <sub>4</sub> .. ..                 | Liquid                                     | " 76°         |
| C <sub>2</sub> Cl <sub>4</sub> O .. .. | Liquid                                     | " 118°        |
| C <sub>2</sub> Cl <sub>6</sub> .. ..   | Solid                                      | " 182°        |
| POCl <sub>3</sub> .. ..                | Liquid                                     | " 110°        |
| PCl <sub>5</sub> .. ..                 | Solid                                      | " 148°        |
| CO <sub>2</sub> .. ..                  | Gas                                        | " -78°        |
| CCl <sub>4</sub> .. ..                 | Liquid                                     | " +76°        |

CuO, CuCl<sub>2</sub>, CuF<sub>2</sub>.—We have (Cu<sub>2</sub>, Cl) = 25.8, Cu<sub>2</sub>, O<sub>3</sub> = 18.6. So that Cl is more strongly attracted by Cu than is O.

So also the fluorides of Cu are very much more unstable than the chlorides, CuF<sub>2</sub>.2H<sub>2</sub>O, for example, spontaneously decomposing in three or four days.

HgO, HgCl<sub>2</sub>, HgF<sub>2</sub>.—The oxides of Hg are less stable than the chlorides. For example, HgCl<sub>2</sub> can be vaporised without decomposition, whereas HgO easily decomposes when heated. The same appears from their heats of formation:—



Also the fluorides of Hg are much less stable than the chlorides, both towards the action of water and of heat.

Thus the hydrated fluoride is decomposed at only 30°, and HgF<sub>2</sub> is decomposed at ordinary temperatures by H<sub>2</sub>O. HgF<sub>2</sub> is decomposed to Hg and F when heated above 200°.

2. The following examples show that when the oxides are more stable than the chlorides, the fluorides are also more stable than the chlorides.

TABLE III.—Showing that the Metallic Fluorides are usually less Fusible than the corresponding Chlorides.

| Compound.               | Physical state. | Fusibility. Melting-point. |
|-------------------------|-----------------|----------------------------|
| LiF .. ..               | Solid           | 801°                       |
| LiCl .. ..              | "               | 598°                       |
| NaF .. ..               | "               | 902°                       |
| NaCl .. ..              | "               | 772°                       |
| KF .. ..                | "               | 789°                       |
| KCl .. ..               | "               | 738°                       |
| RbF .. ..               | "               | 753°                       |
| RbCl .. ..              | "               | 719°                       |
| CuF .. ..               | "               | 908°                       |
| CuCl .. ..              | "               | 434°                       |
| AgF .. ..               | "               | 435°                       |
| AgCl .. ..              | "               | 457°                       |
| CaF <sub>2</sub> .. ..  | "               | 902°                       |
| CaCl <sub>2</sub> .. .. | "               | 719°                       |
| SnF <sub>2</sub> .. ..  | "               | 902°                       |
| SnCl <sub>2</sub> .. .. | "               | 825°                       |
| BaF <sub>2</sub> .. ..  | "               | 908°                       |
| BaCl <sub>2</sub> .. .. | "               | 860°                       |
| MgF <sub>2</sub> .. ..  | "               | 908°                       |
| MgCl <sub>2</sub> .. .. | "               | 708°                       |
| ZnF <sub>2</sub> .. ..  | "               | 734°                       |
| ZnCl <sub>2</sub> .. .. | "               | 262°                       |
| CdF <sub>2</sub> .. ..  | "               | 520°                       |
| CdCl <sub>2</sub> .. .. | "               | 541°                       |
| SbF <sub>3</sub> .. ..  | "               | 296°                       |
| SbCl <sub>3</sub> .. .. | "               | 73.2°                      |
| ZrF <sub>4</sub> .. ..  | "               | White heat                 |
| ZrCl <sub>4</sub> .. .. | "               | 400°                       |

SO<sub>3</sub>, SCl<sub>6</sub>, SF<sub>6</sub>.—SO<sub>3</sub> is a quite stable substance at ordinary temperatures, and only decomposes at a red heat. SCl<sub>6</sub> is at ordinary temperatures too unstable to exist, even SCl<sub>4</sub> only exists below -20°. SF<sub>6</sub> is like SO<sub>3</sub> quite stable even at a red heat.

B<sub>2</sub>O<sub>3</sub>, BCl<sub>3</sub>, BF<sub>3</sub>.—We have (B<sub>2</sub>, O<sub>3</sub>) = 52.8 (B<sub>2</sub>, Cl) = 34.6, showing that B<sub>2</sub>O<sub>3</sub> is very much more stable than BCl<sub>3</sub>. A comparison of the chemical behaviour of the two substances confirms this conclusion. BCl<sub>3</sub>, for example, is easily decomposed by heat and various chemical reagents, such as water; B<sub>2</sub>O<sub>3</sub>, however, is stable at the highest temperatures, and is not decomposed by heating with carbon.

Similarly, BF<sub>3</sub> is very much more stable than BCl<sub>3</sub>. It is not (like BCl<sub>3</sub>) decomposed by electric sparks, nor by Fe at a red heat.

Al<sub>2</sub>O<sub>3</sub>, AlCl<sub>3</sub>, AlF<sub>3</sub>.—Al<sub>2</sub>O<sub>3</sub> is very much more stable than AlCl<sub>3</sub>. Also AlF<sub>3</sub> is very much more stable than AlCl<sub>3</sub> being, unlike the latter, unacted upon by air, acids, or alkalis. AlF<sub>3</sub> resembles Al<sub>2</sub>O<sub>3</sub> rather than AlCl<sub>3</sub> in appearance, properties, and stability.

Mn<sub>2</sub>O<sub>3</sub>, MnCl<sub>3</sub>, MnF<sub>3</sub>.—Mn<sub>2</sub>O<sub>3</sub> and MnF<sub>3</sub> are both, at ordinary temperatures, perfectly stable bodies. MnCl<sub>3</sub>, however, is excessively unstable, spontaneously decomposing at ordinary temperatures into MnCl<sub>2</sub> and Cl.

The first question we have proposed must therefore be answered in the affirmative, namely, fluorine resembles oxygen more closely than does chlorine, because the fluorine atom attracts a given radicle with an intensity of force which approaches nearer that which oxygen exerts on the same radicle than does that which chlorine exerts.

And therefore that the chemical similarity of two elements arises from the fact that both attract the same radicles with nearly the same intensity of force.

This latter conclusion I have confirmed in the case of almost every set of elements in the periodic table which



TABLE IV.—*Showing that Metallic Oxides are usually less Volatile and Fusible than the corresponding Chlorides.*  
(Data largely taken from a compilation by HENRY, *loc. cit.*).

| Compound.                                | Physical state. | Fusibility.                                 | Volatility.                                              |
|------------------------------------------|-----------------|---------------------------------------------|----------------------------------------------------------|
| NbCl <sub>5</sub> .. .. .                | Solid           | Fuses at 194°                               | Boils at 240·5°.                                         |
| Nb <sub>2</sub> O <sub>5</sub> .. .. .   | "               | Infusible                                   | Fixed.                                                   |
| TaCl <sub>5</sub> .. .. .                | "               | Fuses at 211·3°                             | Boils at 241·6°.                                         |
| Ta <sub>2</sub> O <sub>5</sub> .. .. .   | "               | Infusible                                   | Fixed.                                                   |
| Al <sub>2</sub> Cl <sub>6</sub> .. .. .  | "               | Very fusible                                | Boils at 180°.                                           |
| Al <sub>2</sub> O <sub>3</sub> .. .. .   | "               | Fusible in OH flame                         | Fixed.                                                   |
| FeCl <sub>3</sub> .. .. .                | "               | Fuses at 306°                               | Easily volatile.                                         |
| Fe <sub>2</sub> O <sub>3</sub> .. .. .   | "               | Infusible                                   | Fixed.                                                   |
| CrCl <sub>3</sub> .. .. .                | "               | —                                           | Volatile.                                                |
| Cr <sub>2</sub> O <sub>3</sub> .. .. .   | "               | Infusible                                   | Fixed.                                                   |
| CrO <sub>2</sub> Cl <sub>2</sub> .. .. . | Liquid          | —                                           | Boils at 118°.                                           |
| CrO <sub>3</sub> .. .. .                 | Solid           | Fuses about 300°                            | Decomposes.                                              |
| WCl <sub>5</sub> .. .. .                 | "               | Fuses at 248°                               | Boils at 275·6°.                                         |
| WCl <sub>6</sub> .. .. .                 | "               | " 275°                                      | " 346·7°.                                                |
| WCl <sub>6</sub> O .. .. .               | "               | " 210·4°                                    | " 127·5°.                                                |
| WO <sub>3</sub> .. .. .                  | "               | " a red heat                                | Fixed.                                                   |
| VCl <sub>4</sub> .. .. .                 | Liquid          | Does not solidify at -18°                   | Boils at 154°.                                           |
| VO <sub>2</sub> .. .. .                  | Solid           | —                                           | —                                                        |
| VOCl <sub>3</sub> .. .. .                | Liquid          | Does not solidify at -15°                   | Boils at 126·7°.                                         |
| TeCl <sub>4</sub> .. .. .                | Solid           | Fuses at 224°                               | Boils at 414°.                                           |
| TeO <sub>2</sub> .. .. .                 | "               | Fusible                                     | Less volatile than tellurium.                            |
| AsCl <sub>3</sub> .. .. .                | Liquid          | Does not solidify at -29°                   | Boils at 134°.                                           |
| As <sub>2</sub> O <sub>3</sub> .. .. .   | Solid           | Volatile without fusion                     | " 200°.                                                  |
| SbCl <sub>3</sub> .. .. .                | "               | Fuses at 73·2°                              | Boils at 225°.                                           |
| Sb <sub>2</sub> O <sub>3</sub> .. .. .   | "               | " red heat                                  | Volatile below 156°.                                     |
| SbCl <sub>5</sub> .. .. .                | Liquid          | Solid at 0°                                 | Volatilises with SbCl <sub>3</sub> and Cl <sub>2</sub> . |
| Sb <sub>2</sub> O <sub>5</sub> .. .. .   | Solid           | Non-fusible                                 | Non-volatile.                                            |
| BiCl <sub>3</sub> .. .. .                | "               | Fuses at 227°                               | Boils at 427—439°.                                       |
| Bi <sub>2</sub> O <sub>3</sub> .. .. .   | "               | Fusible                                     | Fixed.                                                   |
| HgCl <sub>2</sub> .. .. .                | "               | Fuses at 265°                               | Boils at 295°.                                           |
| HgO .. .. .                              | "               | Infusible                                   | Decomposed at red heat.                                  |
| Hg <sub>2</sub> Cl <sub>2</sub> .. .. .  | "               | Sublimes at 400—500° without melting        | —                                                        |
| Hg <sub>2</sub> O .. .. .                | "               | Infusible                                   | Decomposes.                                              |
| Cu <sub>2</sub> Cl <sub>2</sub> .. .. .  | "               | Fuses at 434°                               | Boils at 954—1032°.                                      |
| Cu <sub>2</sub> O .. .. .                | "               | " a red heat                                | Fixed.                                                   |
| CuCl <sub>2</sub> .. .. .                | "               | Fuses at 498°                               | Decomposes.                                              |
| CuO .. .. .                              | "               | Fuses at bright red heat with decomposition | "                                                        |
| PbCl <sub>2</sub> .. .. .                | "               | Fuses at 498°                               | Boils at 861—954°.                                       |
| PbO .. .. .                              | "               | Fuses about a red heat                      | Volatile at white heat.                                  |
| AgCl .. .. .                             | "               | Fuses at 451°                               | —                                                        |
| Ag <sub>2</sub> O .. .. .                | "               | Infusible                                   | Decomposes.                                              |
| CaCl <sub>2</sub> .. .. .                | "               | Fuses at 719°                               | —                                                        |
| CaO .. .. .                              | "               | Infusible                                   | Fixed.                                                   |
| SnCl <sub>2</sub> .. .. .                | "               | Fuses at 825°                               | —                                                        |
| SnO .. .. .                              | "               | Infusible                                   | Fixed.                                                   |
| BaCl <sub>2</sub> .. .. .                | "               | Fuses above 860°                            | —                                                        |
| BaO .. .. .                              | "               | Fusible in OH flame                         | Fixed.                                                   |
| MgCl <sub>2</sub> .. .. .                | "               | Melts at 708°                               | Volatile.                                                |
| MgO .. .. .                              | "               | Infusible                                   | Fixed.                                                   |
| ZnCl <sub>2</sub> .. .. .                | "               | Fuses at 262°                               | Boils at 676—683°.                                       |
| ZnO .. .. .                              | "               | Infusible                                   | Fixed.                                                   |
| CdCl <sub>2</sub> .. .. .                | "               | Fuses at 541°                               | Boils at 861—954°.                                       |
| CdO .. .. .                              | "               | Infusible                                   | Fixed.                                                   |
| UCl <sub>4</sub> .. .. .                 | "               | —                                           | Volatile.                                                |
| UO <sub>2</sub> .. .. .                  | "               | Infusible                                   | Fixed.                                                   |

betray a striking resemblance to each other. But the results of this research will be set forth elsewhere.

B. We now proceed to deal with the second question.

The accompanying tables show that the corresponding fluorides and oxides approach each other more closely as regards volatility than do the corresponding chlorides and oxides:—

Table I. shows that non-metallic fluorides are more volatile than the non-metallic chlorides.

Table II. shows that the non-metallic oxides are more volatile than the non-metallic chlorides.

Table III. shows that metallic fluorides are usually less volatile than the corresponding metallic chlorides.

Table IV. shows that the metallic oxides are also less volatile than the corresponding metallic chlorides.

In other words, those compounds which approach each other nearest in the intensity of the force with which the atoms are attracted together in the molecule (*viz.*, the oxides and fluorides) are precisely those which approach each other nearest as regards volatility and fusibility.

That is to say, it is the intensity of the internal atomic forces with which the atoms are attracted together in the molecule which determines the intensity of the external attractive force between molecule and molecule, and therefore the volatility of the compound.

This seems a very important conclusion.

A wider research embracing the compounds of all the elements has convinced me that this law is true in its generality. But the results of this research must also be set forth elsewhere.

University of Kiel, December 26, 1903.

## USE OF BISMUTH AS A MEANS OF SEPARATION IN THE RARE EARTH SERIES.

By G. URBAIN and H. LACOMBE.

IN a preceding research (*Comptes Rendus*, vol. cxxxvii., p. 792) we established the fact that bismuth, when employed in the fractionation of double magnesium nitrates, acts as a separating element between samarium and gadolinium. M. Demarçay's europium exists only in minute quantities as compared with gadolinium and samarium in the rare earths, but we have now been able to assign a place to this element in the separation. In our first experiments, however, we started with too small a quantity of material.

Since then we have repeated the experiments with large quantities of oxide. We find that by examination of the absorption spectrum of europium and of its spark spectrum, bismuth inserts itself exactly between samarium and europium.

Bismuth therefore apparently divides the two large groups of the rare earths, and from this we see that europium can be considered as the first term of the series of the yttria earths.

We have therefore utilised, for a new type of separation, the property of the isomorphism of bismuth magnesium nitrate with the same salt of the rare earths. The principle of this method is deduced from the following facts:—

When a series of isomorphous salts give non-crystallisable mother-liquors, the proportion of salt capable of crystallisation retained in the mother-liquors depends on the mass of the non-crystallisable salt accompanying it. Reciprocally, when a large quantity of salt capable of crystallisation is in the presence of a small quantity of non-crystallisable salt in a state of purity, a measurable quantity of this latter is retained in the crystallisation, *a fortiori*, very soluble salts, but nevertheless susceptible of separate crystallisation. Thus the non-crystallisable mother-liquor of magnesium nitrate always retains a proportion of gadolinia, which can be as much as a third of the contained earths. Demarçay considered the fractionation of the

double sodium sulphates to eliminate yttria as one of the most satisfactory methods.

In our method we do not require the multiple treatments which the above method of fractionation necessitates.

If the same weight of magnesium nitrate of bismuth is added to the non-crystallisable mother-liquors, the added salt retains the gadolinium during its crystallisation. The operation should be repeated until the soluble earths give no spark spectrum of gadolinium.

This method of retention recalls the method proposed by Auer von Welsbach to extract from neodymium the last traces of praseodymium by the addition of pure lanthana. But if, in our separation, the bismuth plays an analogous rôle to that of the lanthanum in Auer von Welsbach's method, it is not so inconvenient, since the bismuth can with such great facility be separated from the rare earths in the subsequent operations.

This method of extraction of the gadolinium earths can equally well be used to eliminate gadolinium from crude yttria earths in which this element is present only in small proportion, as in the earth xenotime.

The methodical fractionation of the bismuthic gadoliniferous nitrates in the same order in which they have been obtained allows of the rigorous separation of the ceric and yttric earths. The insolubility of the magnesium nitrates of the earths which precede samarium in the solution of the double nitrate of bismuth allow of the separation of these earths from soluble samaria with an almost quantitative yield.—*Comptes Rendus*, vol. cxxxviii., p. 84.

## LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING DECEMBER 31ST, 1903.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,  
Water Examiner, Metropolis Water Act, 1871.

London, January 9th, 1904.

SIR,—Of the 203 samples examined by us during the month, all were clear, bright, and well filtered.

The rainfall at Oxford during December shows a considerable falling off. The amount of rain actually measured is 0.97 inch; the average fall for the month is 2.16 inches. Thus we have a deficit of 1.19 inch, which, if deducted from the previous excess of 11.74 inches, leaves a total excess for the year of 10.55 inches, or 41.7 per cent on the thirty-five years' average.

TABLE I.—Rainfall in Inches at Oxford, Month by Month, during the Year 1903.

|              | Actual fall.<br>Inches. | Mean of 35 years.<br>Inches. | Difference from the mean. |         |
|--------------|-------------------------|------------------------------|---------------------------|---------|
|              |                         |                              | Inches.                   | Inches. |
| January ..   | 2.58                    | 2.08                         | —                         | +0.50   |
| February ..  | 0.79                    | 1.80                         | −1.01                     | —       |
| March ..     | 3.03                    | 1.47                         | —                         | +1.56   |
| April ..     | 2.28                    | 1.61                         | —                         | +0.67   |
| May ..       | 4.38                    | 1.75                         | —                         | +2.63   |
| June ..      | 5.58                    | 2.10                         | —                         | +3.48   |
| July ..      | 3.47                    | 2.49                         | —                         | +0.98   |
| August ..    | 3.34                    | 2.38                         | —                         | +0.96   |
| September .. | 1.54                    | 2.39                         | −0.85                     | —       |
| October ..   | 6.41                    | 2.76                         | —                         | +3.65   |
| November ..  | 1.47                    | 2.30                         | −0.83                     | —       |
| December ..  | 0.97                    | 2.16                         | −1.19                     | —       |
|              | 35.84                   | 25.29                        | −3.88                     | +14.43  |

Our bacteriological examinations of 366 samples taken during last month have given the results recorded in the

TABLE II.—Showing the Average Monthly Bacterial Variations during the Year 1903.

| Month.          | Thames, derived Companies, unfiltered. | Thames, derived Companies, filtered. | New River, unfiltered. | New River, filtered. | River Lea, unfiltered. | River Lea, (East London), filtered. |
|-----------------|----------------------------------------|--------------------------------------|------------------------|----------------------|------------------------|-------------------------------------|
| January .. ..   | 13161                                  | 69                                   | 319                    | 13                   | 313                    | 38                                  |
| February .. ..  | 15653                                  | 23                                   | 455                    | 8                    | 254                    | 10                                  |
| March .. ..     | 9337                                   | 27                                   | 335                    | 11                   | 582                    | 29                                  |
| April .. ..     | 2820                                   | 15                                   | 149                    | 6                    | 193                    | 20                                  |
| May .. ..       | 4625                                   | 19                                   | 231                    | 11                   | 351                    | 26                                  |
| June .. ..      | 10337                                  | 42                                   | 151                    | 7                    | 474                    | 28                                  |
|                 | 9322                                   | 32                                   | 273                    | 9                    | 361                    | 25                                  |
| July .. ..      | 3805                                   | 23                                   | 258                    | 11                   | 261                    | 36                                  |
| August .. ..    | 109 0                                  | 43                                   | 270                    | 15                   | 189                    | 33                                  |
| September .. .. | 6201                                   | 39                                   | 333                    | 19                   | 330                    | 29                                  |
| October .. ..   | 16671                                  | 66                                   | 843                    | 30                   | 470                    | 25                                  |
| November .. ..  | 18390                                  | 43                                   | 845                    | 25                   | 610                    | 28                                  |
| December .. ..  | 27216                                  | 37                                   | 861                    | 40                   | 226                    | 18                                  |
|                 | 13866                                  | 41                                   | 563                    | 23                   | 347                    | 28                                  |
|                 | 11594                                  | 36                                   | 420                    | 16                   | 354                    | 26                                  |

Mean of 1st 6 months.

Mean of 2nd 6 months.

Mean of 12 months.

TABLE III.—Average Number of Microbes in the Filtered London Waters for the Past Seven Years.

|            | Thames-derived Companies. | New River. | River Lea. |
|------------|---------------------------|------------|------------|
| 1897 .. .. | 46                        | 32         | 62         |
| 1898 .. .. | 34                        | 30         | 25         |
| 1899 .. .. | 27                        | 15         | 20         |
| 1900 .. .. | 21                        | 9          | 10         |
| 1901 .. .. | 24                        | 10         | 22         |
| 1902 .. .. | 27                        | 13         | 25         |
| 1903 .. .. | 36                        | 16         | 26         |

TABLE IV.—Averages of the Supplies derived from the River Thames during the Year 1903.

|                 | Common salt per gallon. Means. | Nitric acid per gallon. Means. | Hardness. Degrees. Means. | Oxygen required per gallon. Means. | Organic carbon per gallon. Means. | Organic carbon per gallon. Maxima. | Colour. Brown : Blue. Means. |
|-----------------|--------------------------------|--------------------------------|---------------------------|------------------------------------|-----------------------------------|------------------------------------|------------------------------|
| January .. ..   | 2 335                          | 1'149                          | 16 56                     | 0'064                              | 0'191                             | 0'293                              | 25'5 : 20                    |
| February .. ..  | 2 391                          | 1'040                          | 17'07                     | 0'056                              | 0'165                             | 0'255                              | 20'5 : 20                    |
| March .. ..     | 2 365                          | 0 911                          | 15'96                     | 0'063                              | 0'190                             | 0'281                              | 21'9 : 20                    |
| April .. ..     | 2'204                          | 0'779                          | 14'77                     | 0'050                              | 0'154                             | 0'250                              | 14 6 : 20                    |
| May .. ..       | 2'083                          | 0 701                          | 14'29                     | 0'066                              | 0'192                             | 0'299                              | 19'5 : 20                    |
| June .. ..      | 1 919                          | 0 605                          | 14 13                     | 0'062                              | 0'189                             | 0 338                              | 23 4 : 20                    |
| July .. ..      | 1'956                          | 0'700                          | 15'61                     | 0'066                              | 0'155                             | 0'251                              | 20'6 : 20                    |
| August .. ..    | 2'029                          | 0'626                          | 15'16                     | 0'054                              | 0'148                             | 0'194                              | 14'6 : 20                    |
| September .. .. | 2'166                          | 0'707                          | 15 68                     | 0'055                              | 0'164                             | 0'246                              | 14'2 : 20                    |
| October .. ..   | 2'019                          | 0'731                          | 15'08                     | 0'083                              | 0'229                             | 0'400                              | 26'8 : 20                    |
| November .. ..  | 1 957                          | 0'739                          | 16 78                     | 0' 88                              | 0'245                             | 0 388                              | 29 8 : 20                    |
| December .. ..  | 2'029                          | 1'025                          | 16'74                     | 0' 66                              | 0 205                             | 0'244                              | 22 3 : 20                    |

following table. Besides these samples we have examined 362 others from special wells, standpipes, &c., making 728 samples in all :—

|                                                                                                           | Microbes per c.c. |
|-----------------------------------------------------------------------------------------------------------|-------------------|
| New River, unfiltered (mean of 25 samples) ..                                                             | 861               |
| New River, filtered (mean of 75 samples) ..                                                               | 40                |
| Thames, unfiltered (mean of 24 samples) ..                                                                | 27,216            |
| Thames-derived water from the clear-water wells of eight Thames-derived supplies (mean of 194 samples) .. | 37                |
| Ditto ditto .. .. highest                                                                                 | 376               |
| Ditto ditto .. .. lowest                                                                                  | 0                 |
| River Lea, unfiltered (mean of 24 samples) ..                                                             | 226               |
| River Lea, from the East London Water Company's clear-water wells (mean of 24 samples)                    | 18                |

Of the 293 daily samples taken from the general filter wells of the Metropolitan Water Companies, sixteen samples, or 5·4 per cent, were sterile. Thirty-one samples, or 10·5 per cent, contained more than 100 microbes, and of these, nine samples contained more than 150 microbes per c.c. The thirty-one excess samples contained an

average of 156 microbes per c.c. In November thirty samples contained an average of 168 microbes per c.c.

The above figures show that the bacterial condition of the unfiltered water was worse than in November, but that of the filtered supply was much better.

During the past year we have examined 8022 samples of London waters bacteriologically, as compared with 6953, 6893, and 6731 respectively in the three previous years. We have also made 2614 chemical analyses of London waters throughout this period, making a total of 10,636 samples examined during the year.

Table II. gives the bacteriological results for the past year. During the first six months the Thames-derived Companies' clear-water wells contained on an average 32 bacteria per c.c., while the New River and River Lea supplies contained respectively 9 and 25. During the second six months the numbers of bacteria in the waters from the same three sources were 41, 23, and 28 respectively.

Table IV. gives the average chemical composition of the Thames-derived supplies during the year 1903. The relatively exceptional high values of the organic carbon during the autumn months is to be attributed to the greatly excess-

sive rainfall, causing the solution of considerable amounts of vegetable matter, naturally associated with an increase of colour in the supply. Considering the abnormal meteorological conditions, the filtration of the London waters has been on the whole successfully carried out. So far as the Returns of the Registrar-General show, the conditions of Public Health have been highly satisfactory, and it is interesting to note that there has been no suggestion of the occurrence of any water-borne disease.

We are, Sir,  
Your obedient Servants,  
WILLIAM CROOKES.  
JAMES DEWAR.

### REPORT ON ASH.\*

By G. S. FRAPS.

(Concluded from p. 43).

#### Work of H. C. Sherman on Sulphur and Phosphorus.

H. C. SHERMAN, of Columbia University, New York City, during the past summer has been studying the methods for the determination of sulphur and phosphorus in organic materials. We have had some correspondence on the subject, and he has kindly communicated to me the results of his work, which will appear in the *Journal of the American Chemical Society*. The methods which Dr. Sherman has studied are as follows:—

1. Combustion in oxygen under pressure of 25 to 30 atmospheres in the bomb calorimeter.

2. Fusion with alkali, as described by Osborne.

3. The provisional method of this Association (the nitric acid method), with the use of 2 grms. of sample.

Six samples of material were used. In every case the nitric acid method gave lower results than the other two methods, the average amount of sulphur found being about five-sixths of that present. It seems probable that the greater part of the discrepancy is due to volatilisation of sulphur in some form of combination not sufficiently oxidised to stand the heat generated at this point. Such a loss could doubtless be avoided by repeated evaporation with nitric acid, or by the addition of alkali along with the nitrate, but the method would thus become practically that of Salkowski. Combustion in oxygen has the advantage over the fusion method.

The fusion method is described by Osborne as follows (*Journ. Am. Chem. Soc.*, 1902, xxiv., 142):—

About 10 grms. of sodium peroxide were converted into hydroxide in a nickel crucible by adding a little water and boiling over an alcohol lamp until the excess of water was expelled. From 1 to 2 grms. of the protein were then stirred into the slightly cooled hydroxide, and oxidised by gradually raising the heat and adding small portions of sodium peroxide until the oxidation was complete. The fused mass was then dissolved in 400 c.c. of water, its solution strongly acidified with hydrochloric acid, boiled until the excess of peroxide was destroyed and chlorine expelled, filtered, made neutral with ammonia, and an excess of 4 c.c. of concentrated hydrochloric acid added. From the boiling solution sulphuric acid was precipitated by gradually adding a solution containing 1 gm. of barium chloride. After standing overnight on a steam table the barium sulphate was filtered out, washed, ignited, and weighed.

Dr. Sherman uses 15 grms. of sodium peroxide, and with vegetable materials the acidulated solution was evaporated to dryness, and heated to dehydrate any silica which might have been present.

This method differs from that tested by the referee chiefly in the use of a larger volume of water in the solution from which the barium sulphate was precipitated.

As it is well known that barium sulphate is soluble in strong solutions of salts, it is possible that this may account for the difference.

Dr. Sherman exchanged samples with the referee, with the following results. The sample of lean meat was sent by Dr. Sherman. The other two samples are the referee's samples:—

TABLE V.—Comparison of Methods for the Determination of Sulphur.

| Method and analyst.              | Lean meat.  | Cotton-seed meal.          | Corn bran.                 |
|----------------------------------|-------------|----------------------------|----------------------------|
|                                  | Per cent S. | Per cent SO <sub>3</sub> . | Per cent SO <sub>3</sub> . |
| Compressed oxygen method—Sherman | 0.82        | 1.49                       | 0.47                       |
| Fusion method—Sherman .. ..      | 0.81        | —                          | —                          |
| Nitric acid method—Sherman .. .. | 0.68        | —                          | —                          |
| Nitric acid method—Fraps .. ..   | 0.57        | 1.03                       | 0.40                       |
|                                  | 0.52        | 1.07                       | 0.41                       |

I am convinced by this work that the nitric acid method gives too low results.

#### Determination of Potash.

The results obtained by the different analysts for potash by the methods already described are as follows:—

TABLE VI.—Potash Determinations.

| Analyst.       | Corn bran.                |                                     | Cotton-seed meal.         |                                     |
|----------------|---------------------------|-------------------------------------|---------------------------|-------------------------------------|
|                | Method I. Sulphuric acid. | Method II. Charring and extracting. | Method I. Sulphuric acid. | Method II. Charring and extracting. |
|                | Per cent.                 | Per cent.                           | Per cent.                 | Per cent.                           |
| J. H. Gibboney | 0.43                      | 0.39                                | 1.80                      | 1.82                                |
|                | 0.42                      | 0.39                                | 1.78                      | 1.82                                |
| E. G. Runyan   | 0.37                      | 0.38                                | 1.61                      | 1.73                                |
|                | 0.38                      | 0.36                                | 1.56                      | 1.68                                |
|                | —                         | —                                   | 1.60                      | 1.72                                |
|                | —                         | —                                   | 1.64                      | 1.77                                |
| R. W. Thatcher | 0.42                      | 0.37                                | 1.74                      | 1.64                                |
|                | 0.45                      | 0.38                                | —                         | 1.72                                |

There seems to be a slightly larger quantity of potash obtained by charring and extracting than by ignition with sulphuric acid, though the results are not conclusive.

#### Determination of Sulphates.

It has been suggested to the referee that not the total sulphur but the sulphur existing as sulphates in plants should be considered as the sulphur of the ash, since the organic sulphur would otherwise be counted twice. It is necessary, however, that we should have a method for the determination of total sulphur, in order to estimate the quantity of that element required by the growing plant; the fact should not be lost sight of for an instant that the determination of sulphur in an ash, as heretofore made, throws no light whatever upon the quantity of sulphur in the plant, and, since it leads to false conclusions, is worse than valueless.

That part of the sulphur in plants exists as sulphates, and part in the organic form, has been known for a long time. R. Arendt, in 1856, was the first to distinguish between organic and inorganic sulphur in plants, and to estimate total sulphur with any degree of accuracy. He extracted the sulphates with acidulated water, and estimated the total sulphur by fusion with alkalis. Ulrich pursued a similar plan. Arendt (1857) found small amounts (0.04–0.06 per cent) of sulphates in the lower leaves and ears of the oats, and more in the upper leaves (0.22–0.44 per cent). Ulrich (1859) states that sulphates are totally absent from the lower leaves and stems of red clover, while present in the upper leaves and the blossom.

Wolff and his associates (1859) give the following

\* Proceedings of the Nineteenth Annual Convention of the Association of Official Agricultural Chemists, held at Washington, D.C.

quantity of sulphates and organic sulphur as present in the dry matter of green rape.

TABLE VII.—Distribution of Sulphur in Green Rape (Wolff).

| Part of plant. | Organic sulphur.<br>Per cent S. | Sulphates.<br>Per cent SO <sub>3</sub> . |
|----------------|---------------------------------|------------------------------------------|
| Seeds .. .. .  | 1.127                           | 0.020                                    |
| Straw .. .. .  | 0.167                           | 0.021                                    |
| Chaff .. .. .  | 0.310                           | 1.464                                    |

Wolff and Yelin (1860) did not find any sulphates in the grain or straw of wheat, in barley, oats, or lucerne. Knop and Ritter (1859) found sulphates in garden beans (0.14 per cent), but none in the bean straw.

E. Schulze (*Landw. Versuchs. Stat.*, 1876, xix., 172) investigated the changes in sulphur during the sprouting of the seeds of the yellow lupine. The finely ground material was completely extracted with warm water, acidified with hydrochloric acid after separating a small quantity of albumen, and precipitated with barium chloride. As the precipitate did not appear pure it was fused with sodium carbonate with the addition of a little potassium nitrate, the melt extracted with water, and the sulphuric acid precipitated with barium chloride. The seed contained 1.028 per cent sulphur, 0.154 per cent as sulphates (0.385 per cent calculated as SO<sub>3</sub>). The sprouted seeds contained 1.510 per cent and 1.703 per cent sulphur as sulphates after twelve and fifteen days respectively. An oxidation of organic sulphur took place during the germination.

Tammann (*Ber.*, 1886, xix., 261, Ref.) obtained 0.359 per cent SO<sub>3</sub> from peas, by fusion with sodium carbonate and saltpetre, and 0.075 per cent SO<sub>3</sub> precipitated by barium hydroxide from the hot water extract purified with tannic acid. After germinating twenty-five days, 0.191 per cent SO<sub>3</sub> was present—a decided increase. By boiling the alkaline filtrate from the barium sulphate with hydrochloric acid a second precipitate appeared, due, he says, to the presence of ethereal salts of sulphuric acid. The quantity of this second precipitate from the ungerminated seeds was insignificant as well as from the seeds germinated in darkness, while from those germinated in the light it corresponded to 0.019 per cent SO<sub>3</sub>.

Berthelot and Andre (*Biedermann's Central-Blatt*, 1891, 555) investigated the sulphur in different forms in plants at different stages of growth. They concluded that the sulphur content of the plant increases continuously until it blossoms. The amount of sulphur present in organic compounds reaches its maximum at the time of flowering, and then gradually decreases partly on account of re-oxidation of the sulphur, partly by elimination of volatile sulphur compounds. The proportion of the two forms of sulphur in the seeds varies considerably in different species of plants. In *Avena sativa* nearly all the sulphur is present in the organic form; on the other hand, in the white lupine only 6.7 per cent of the total sulphur is in organic combination.

I have gone thus fully into the literature in order to give a clear idea as to the present standing of the determination of sulphates in plants. It is seen that the quantity of sulphates present may vary considerably, both in the seeds of different plants, different parts of different plants, different parts of the same plants, and in the same plant at different stages of growth. At times, indeed, the amount of sulphates present as sulphates is very small.

The following method was used for the determination of sulphates in several materials:—

Five grms. substance was mixed well with 50 c.c. of a 1 per cent solution of hydrochloric acid, allowed to stand half-an-hour, filtered, and washed with the dilute acid to 250 c.c. or more. The liquid was heated to boiling, barium chloride was added, and the determination completed in the usual way.

Instead of the 1 per cent hydrochloric acid, water was used in several cases, but it was found to dissolve organic

matter which was afterwards precipitated on coming in contact with the acid.

No sulphur as sulphates was found in the following materials:—Corn, peas, green millet, timothy hay, corn silage, peanuts.

Sulphur as sulphates was found in the following:—Oats (trace); crimson clover straw, 0.003 per cent; cotton-seed meal, 0.001 per cent; green cowpea vines, 0.085 per cent.

#### Determination of Chlorine.

The writer has already called attention to the fact that chlorine as well as sulphur will be lost in burning a plant substance to an ash by itself. It will appear later that the loss is due to decomposition of the chlorides by the heated organic matter.

Chlorine was determined in a number of substances in two ways, namely, in the ash as prepared in the usual way and by fusion of the plant material with sodium hydroxide, a method which will be described in full later on in this paper. The results of the determination are as follows:—

TABLE VIII.—Chlorine in Plants.

| Material.                    | Ash method. | Sodium hydroxide method. |
|------------------------------|-------------|--------------------------|
|                              | Per cent.   | Per cent.                |
| Corn .. .. .                 | trace       | 0.040                    |
| Peas .. .. .                 | 0.005       | 0.008                    |
| Peas, No. 2 .. .. .          | 0.001       | 0.012                    |
| Oats .. .. .                 | 0.005       | 0.097                    |
| Oats, No. 2 .. .. .          | 0.001       | 0.056                    |
| Peanuts .. .. .              | 0.008       | 0.017                    |
| Cotton-seed meal .. .. .     | 0.008       | 0.032                    |
| Cotton-seed hulls .. .. .    | 0.005       | 0.013                    |
| Linseed meal .. .. .         | 0.010       | 0.029                    |
| Millet .. .. .               | 0.120       | 0.187                    |
| Corn silage (dry) .. .. .    | 0.310       | 0.607                    |
| Green rape (dry) .. .. .     | 1.261       | 1.290                    |
| Green peas (dry) .. .. .     | 1.160       | 1.205                    |
| Sweet potatoes (dry) .. .. . | 1.341       | 1.362                    |
| Timothy hay .. .. .          | 1.864       | 1.888                    |

It is evident that a considerable proportion of the chlorine in plants may be lost in burning them to an ash, particularly when there is only a small quantity of chlorine in the plant.

The cause of the loss is shown by the work of Davies, reported in the *Journal of the Society of Chemical Industry*, (1901, xx., 98). He experienced similar losses when sugar, starch, or filter-paper was ignited with sodium chloride. The alkalinity of the ash was evidence that no base had been lost, and if the sodium chloride was added after the sugar had been charred, and the ignition then completed, no loss took place; that is to say, the loss took place during the initial stages of the combustion, and not during the time the carbon was being burned off, and was probably due to the decomposition of the chlorides by the hot vapours from the organic matters. Extracting the material with hot water after charring and then completing the combustion can therefore have no effect on the loss of chlorine, since it takes place before the extraction. When sodium carbonate equivalent to 5 per cent of the organic matter was added to the material, the loss of chlorine was prevented.

Three methods for the determination of chlorine in plants were tested:—

#### 1. Ignition of the Material with Calcium Acetate.

Five grms. substance were moistened with 20 c.c. of a solution of calcium acetate containing 0.56 gm. calcium oxide, evaporated to dryness, and ignited. The ash was dissolved in nitric acid, filtered, and chlorine determined in the filtrate by the Volhard method. On account of the very small quantity of chlorine present in many of the substances, a gravimetric method could not be used, as the precipitate would not coagulate. The results by this method are low (Table IX.).

2. *Fusion with Sodium Hydroxide.*

Five grms. substance were mixed thoroughly with 10 c.c. of a solution of 100 grms. caustic soda in 200 c.c. water, evaporated to dryness, and finally fused. A nickel crucible was used for a portion of the work and silver crucibles for the remainder, due care being taken to prevent silver from being detached from the crucible. It is better to use nickel crucibles, as there is always a little danger of small particles of silver being detached. The melt was dissolved in water, neutralised with nitric acid, filtered, and chlorine determined in the filtrate by the Volhard method.

The method yields satisfactory results, but it is long, and the crucibles wear out rapidly.

3. *Ignition with Sodium Carbonate.*

Five grms. substance in a platinum dish are impregnated with 20 c.c. of a 5 per cent solution of sodium carbonate, evaporated to dryness, and ignited as thoroughly as possible. The residue is extracted with hot water, filtered, and washed. It is returned to the platinum dish, ignited to an ash, dissolved in nitric acid, and the chlorine determined by the Volhard method.

In one or two cases the filtrate from the first extraction was yellow from organic matter. It was returned to the dish with the charred residue, evaporated to dryness, and ignited. The residue burned very readily to a white ash. The sodium carbonate method gives as accurate results as the method of fusion with caustic soda, and as it is shorter and easier, it is to be recommended for use.

The results by the three methods are contained in the following table:—

TABLE IX.—*Methods for Chlorine.*

| Material.         | Ignition with<br>calcium acetate.<br>Per cent. | Fusion with<br>caustic soda.<br>Per cent. | Ignition with<br>sodium carbonate.<br>Per cent. |
|-------------------|------------------------------------------------|-------------------------------------------|-------------------------------------------------|
| Corn .. .. .      | 0.002                                          | 0.040                                     | 0.027                                           |
|                   | —                                              | 0.040                                     | 0.026                                           |
| Millet . . . .    | 0.148                                          | 0.165                                     | 0.188                                           |
|                   | —                                              | 0.171                                     | 0.194                                           |
|                   | —                                              | 0.204                                     | —                                               |
| Corn silage ..    | 0.400                                          | 0.616                                     | 0.610                                           |
|                   | —                                              | 0.599                                     | —                                               |
| Cotton-seed meal  | 0.031                                          | 0.032                                     | 0.020                                           |
| Cotton-seed hulls | 0.010                                          | 0.013                                     | 0.017                                           |
| Green peas ..     | 0.215                                          | 0.197                                     | 0.228                                           |
|                   | —                                              | 0.212                                     | 0.239                                           |
| Oats .. . . .     | 0.034                                          | 0.056                                     | —                                               |
| Timothy hay ..    | 0.870                                          | 0.914                                     | 0.897                                           |
|                   | —                                              | 0.878                                     | 0.911                                           |
|                   | —                                              | 0.872                                     | —                                               |

*Recommendations.*

The referee makes the following recommendations:—

1. Either that the method of fusion with sodium hydroxide and sodium peroxide be adopted as a provisional method by this Association, or else that the referee on ash be requested to make a further study of the fusion method.

2. That the method of determining chlorine by ignition with sodium carbonate be adopted as a provisional method.

A NEW METHOD FOR THE ATTACK OF  
GALENAS AND CHALCOPYRITES.

By C. BOUCHER.

WHEN we have to estimate all the elements of a galena or a chalcopyrites, we often experience much difficulty in finding a sufficiently rapid and complete method for dissolving the mineral. We do not propose here to refer to all the well-known methods for the attack of these minerals, as most of them have come to be, in a way, classic. However, they are not always very convenient to carry out, most of these methods requiring a considerable time. We

think we have overcome this drawback in the method we are about to describe.

We have had recourse to the action of persulphates, salts which easily give up their oxygen, and of which the acid is fairly energetic. A first series of assays by the wet method, which consisted in boiling the sample of mineral with a concentrated solution of persulphate of ammonium, did not give very satisfactory results.

Still, after a certain time, the sulphide of lead of the galena is completely transformed into sulphate; a change which is also brought about, but less quickly, with sulphate of ammonium, as has been shown by Mascazzini.

By adding to the solution a few drops of concentrated nitric acid, the reaction becomes more rapid, and succeeds also with chalcopyrites. In all cases with this latter mineral the formation of spongy sulphur, which takes a long time to get rid off, becomes an annoying obstacle.

It is necessary then to boil for a long time on a sand-bath, to evaporate almost to dryness to melt the sulphur, and then to make certain that the resulting globule of sulphur does not contain any more of the mineral.

A second series of experiments made by the dry way gave excellent results. As reagent we used a mixture of 3 parts of persulphate of sodium\* and 1 part of nitrate of ammonium, more or less approximately. The use of persulphate alone is insufficient, even in the cold galena gives off sulphuretted hydrogen on contact with these salts.

I. *Treatment of Galenas.*

We operate on 1 or 2 grms. of the well-powdered mineral, mixed more or less intimately, by means of a platinum wire, with four or five times its weight of the above-mentioned reagent; these amounts are only approximate. The operation may be performed either in an iron crucible, if the estimation of the iron is not indispensable, or in a sufficiently large porcelain crucible.

The mass must not occupy more than one-third of the crucible, which must be covered. The whole is then placed on a moderately heated sand-bath. After five or six minutes the crucible is removed, allowed to cool, and examined to see that the attack is complete (in this case no more black grains of galena will be visible). It suffices now to take up with water, and filter. On the filter we shall find the lead in the form of sulphate, mixed with the gangue, silica, &c., which are separated by the known methods. As for the sulphuric solution, it contains the other metals.

To better follow the progress of the reaction, and also to avoid all loss, we may very advantageously effect the fusion in an Erlenmeyer flask of Jena glass, about 100 c.c. capacity, for example, taking care to shake from time to time, and not to heat too strongly at the commencement.

II. *Treatment of Chalcopyrites.*

We operate on, say, 1 grm. of the powdered sample, passed through a fine sieve. Mix as above with 4 or 5 parts of the reagent. Place the whole, by preference, in a Kjeldahl matras of Jena glass. The neck of the matras prevents projections, as the reaction is fairly lively. Heat on a sand-bath, or directly over a Bunsen flame. It is most essential to heat very moderately at the commencement, regulating the heat carefully. When the deflagration is over, we may heat more strongly, shaking the matras from time to time so that the fragments thrown up on the sides do not escape the reaction. The heat is continued until the reaction is finished (about five or six minutes); no more black grains should then be seen at the bottom of the matras.†

As soon as the reaction commences the mass takes a greenish-blue colour, due to the formation of sulphate of copper: this is a rapid means for distinguishing between a pyrites and a chalcopyrites. When the attack is

\* Taken in preference to the ammonium salt, as it is more stable. This latter salt, in fact, decomposes fairly rapidly with heat.

† See our remark relative to Mn.

complete, allow to cool by spreading the mass over the sides of the matras. Take up with hot water, heat gently if necessary, and filter to separate the gangue ( $\text{SiO}_2$ ). We obtain thus a sulphuric solution in which the metals can be estimated by the known methods.

It is unnecessary to add that this method requires a separate estimation of the sulphur.

#### Remarks.

If the minerals analysed contain any notable quantity of manganese, we must not forget that this metal will be found in the residue, in the form of  $\text{MnO}_2$ .

We may recall the experiments made by Marshall, who showed that manganese is precipitated from its acid solutions (nitric or sulphuric) in the form of  $\text{MnO}_2$  by boiling persulphates. (See also M. Dittrich and C. Hassel, *Berichte*, 1902, vol. xxxv., p. 3266, on this subject).

If a first tentative attack of the mineral does not succeed completely, we may very well add a further quantity of the reagent. In all cases it is not necessary to attach too much importance to the slight greenish-black residue left by some minerals (especially by chalcopyrites) after this attack, and of which the appearance may be similar to that of the mineral itself. It is generally composed of the unattackable gangue mixed with sulphur. However, for greater security, it is advisable, after taking up the fused and cooled mass with water, to filter rapidly without pouring out the residue, and after washing to treat the latter with a few drops of concentrated nitric acid. This solution, with the wash waters, is added to the principal solution, throwing the final residue on to the filter.

It may be asked, have the persulphates any action on glass? This has not been taken into consideration in view of the short time the reaction lasts. If necessary, platinum vessels could be used instead of glass ones. The truth will be inquired into later on.

As for the other sulphurised minerals, at least the pyrites, blendes, stibines, mispickel, &c., they have not furnished satisfactory results. They appear to be much more stable with the persulphates than are the galenas and chalcopyrites.

A curious thing about these latter is that they are much less easily attacked by acids than the former. Pyrites, especially, almost entirely resists the reagent used above.

Blendes are attacked very incompletely, and the reaction is accompanied with little explosions and the burning of the sulphur.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 18.

## PROCEEDINGS OF SOCIETIES.

### PHYSICAL SOCIETY.

*Ordinary Meeting, January 22nd, 1904.*

Dr. R. T. GLAZEBROOK, F.R.S., President, in the Chair.

MR. W. BENNETT read a paper entitled "*Notes on Non-homocentric Pencils, and the Shadows produced by them.*"  
1. *An Elementary Treatment of the Standard Astigmaticencil.*"

It is shown that several of the properties of the standard astigmatic pencil, and the variations in the form of its cross section, can be simply deduced from a consideration of the projections of its rays upon two planes, each of which is at right angles to one of the two focal lines. The projection of the rays are in each case concurrent. The shadow of a straight wire at right angles to the axis is also dealt with, and it is shown that the rays intercepted by the wire are one set of generators of a hyperbolic paraboloid. The section of this surface by any other plane is a hyperbola or a parabola. The rays are seen to be all parallel to a plane through the axis. If the object wire is not at right angles to the axis the shadow surface is a hyperboloid of

one sheet. The section by any plane is, in general, a hyperbola, which is rectangular when the plane is at right angles to the axis, and reduces to two straight lines when the plane passes through either of the focal lines. The asymptotes of the rectangular hyperbolas lie in two planes which pass respectively through the two focal lines. The author showed string models of the various pencils and shadow surfaces, and of pencils produced by lenses or mirrors. The paper concludes with a simple method for obtaining, by the method of sagittæ, the positions of the approximate lines produced in a small pencil refracted obliquely through a lens.

Prof. S. P. THOMPSON congratulated the author, and said that although many people accustomed to these pencils could easily follow the passage of the rays by the inspection of diagrams, there could be no doubt that there were others to whom the subject could only be made clear by the use of models. Geometrical models had an advantage because they could be so constructed as to show the characteristics of the particular aberration under consideration, a condition of things which could not be realised in the case of actual pencils produced by the passage of light through a lens system. The study of the peculiar shadows produced by these beams was one of the out of the way branches of physics which could be pursued with advantage. The results were so striking that they often engendered a curiosity leading to a further study of optics.

Prof. J. D. EVERETT expressed his interest in the paper and in the way the author had investigated the shadow phenomena by obtaining the equation to the ruled surface generated by the shadow of the rod. He drew attention to another point in the paper. Although a sloping rod generally gives a curved shadow, it is always possible to rotate the screen and get a straight shadow.

Mr. R. J. SOWTER said that in constructing a string model of the standard elliptic-sectioned beam, it was convenient to thread the strings between two lines representing the focal lines, and to obtain the corresponding points in the focal lines by projection from circles, since the points moved along the focal lines according to a simple harmonic motion rule. Further, it was useful to take stereoscopic photographs of the string models, so that the solid effects could be reproduced in the stereoscope, and the models thus replaced by the photographs.

The AUTHOR, in reply to Prof. Thompson, said the geometrical method gave results very simply when the aberration was purely astigmatic, but was less readily applicable when other aberrations were present. The formation of shadows could then be more readily followed by considering the form of the wave front, the way in which it meets the object wire, and the consequent evolutions of the trace left upon it by the object wire. Replying to Mr. Sowter, he said that the general method of constructing the models was simplified in many cases, both end plates being produced from a single easily-constructed template.

A paper by Prof. R. W. WOOD on "*Some New Cases of Interference and Diffraction*" was ready by the SECRETARY.

In this paper Prof. Wood discusses certain types of the interference of light which have been known for many years, as well as some cases which he thinks are quite new. The colours of mixed plates, and the phenomena of interference in transparent films deposited on metallic reflectors are the cases chiefly considered. The facts which have been brought out may be summed up as follows:—The colours of mixed plates are due to diffraction, and should not be classed with interferences in their films. The explanation originally given by Young, and the treatment given by Verdet and others, are unsatisfactory, and do not indicate what becomes of the energy. In the cases of films deposited on perfectly reflecting surfaces, which, according to the elementary theory, should exhibit no interference colours, we may, under certain conditions, have colours far more brilliant and quite as saturated as any shown by the soap bubble. In other cases, where at first sight no interference appears to have

taken place, we may, by employing polarised monochromatic light, obtain fringes of a very curious nature, which are the result of the interference between the elliptical vibration coming from the metal surface and the plane-polarised vibration reflected from the surface of the transparent film. The Secretary showed by means of the lantern the colours produced by reflection from thin films of mica and collodion deposited on silver. He also showed the polarised fringes produced by the interference of two streams of light polarised at right angles. The incident sodium light polarised at an angle of  $45^\circ$  with the plane of incidence was reflected from a wedge-shaped film of gelatin deposited on speculum metal, and the fringes viewed with an analysing nicol.

Mr. JULIUS RHEINBERG said that Prof. Wood's paper had been of special interest to him; in particular as he found in it confirmation of the results afforded by some experiments he had made some time ago. He thought it was not generally known that some striking examples of the colours of mixed plates were to be found in Nature. A few years ago there had been some discussion in the Quekett Microscopical Club as to the origin of certain brilliant colours shown by the diatom *Actinocyclus Ralfsii* when seen with an objective of small numerical aperture. From an optical point of view, the diatom valves in question might be looked upon as plates of colourless siliceous material perforated by numerous tiny holes, and he had explained the matter as being a natural case of the colours of mixed plates. Mr. Rheinberg exhibited a number of specimens of *Actinocyclus Ralfsii* under the microscope.

A paper by Mr. S. SKINNER "On the Photographic Action of Radium Rays" was taken as read.

It is well known that a photographic plate by exposure to radium rays is affected in such a way that the plate develops similarly to its development after exposure to light. The experiments described in the paper are an attempt to answer the question: Are the actions the same? As far as can be seen, the final results of the actions and developments are the same, and the experiments appear to indicate that only slight differences occur in the early stages. The plates, suitably enclosed, were subjected to the rays from varying quantities of radium bromide for varying times, and the results showed that the intensity of the developed image increased rapidly to a maximum value with increase of exposure, then decreased, first rapidly, and finally very slowly until a stage was reached in which there was practically no dark image formed on development. It is found that spark images are at first obliterated by radium rays which do not cause such a great density as that of the spark images obliterated, and that with prolonged exposure the radium rays reverse the spark images.

Mr. W. A. PRICE exhibited a number of instruments constructed by Messrs. Crompton and Company.

The instruments shown were chiefly switches, galvanometers, and potentiometers, and Mr. Price explained that the chief interest of the exhibit lay in the mechanical devices which had been introduced to overcome difficulties of design.

Royal Institution.—On Thursday next, February 4, Mr. A. D. Hall, at 5 o'clock, delivers the first of three lectures at the Royal Institution on "Recent Research in Agriculture"; and on Saturday, February 6, at 3 o'clock, Mr. Charles Waldstein commences a course of two lectures on (1) "The Study of Style in Greek Sculpture," (2) "Culture and Sculpture"; and on Saturday, February 20, Lord Rayleigh begins his course of six lectures on the "Life and Work of Stokes." The Friday Evening Discourse on February 5 will be delivered by Mr. Alfred Austin, the Poet Laureate, his subject being "The Growing Distance for the Higher Kinds of Poetry; the discourse on February 12 will be delivered by Dean Robinson on "Westminster Abbey in the Early Part of the Seventeenth Century"; and on February 19 by Mr. C. T. R. Wilson on "Condensation Nuclei."

## CORRESPONDENCE.

### WHY ARE SOME ELEMENTS SO MUCH MORE ABUNDANT THAN OTHERS?

To the Editor of the Chemical News.

SIR,—With regard to the suggestion of your correspondent, Mr. Geoffrey Martin, that the varying abundance of the elements may be due to the decay of the less stable, is not our actual knowledge of the material of the universe at present too limited to admit of any satisfactory attempt to answer this question? Is not our real knowledge of matter limited to a mere egg-shell crust of this planet, and the outer envelopes of the sun and stars? If the earth were once in a fluid state, it seems reasonable to suppose that the heavier elements would seek the centre of the mass. May it not well be that the core of the planet consists of gold and platinum, osmium and iridium, while further from the centre there may exist a layer of uranium and radium, tungsten, and so on, the small quantities of these elements found at the surface having been thrown up in the tremendous internal commotions which must have been constantly occurring, similar to the disturbances in the sun which produce sun-spots at present? The principal elements detected in the sun's surface are amongst those of least specific gravity and greatest volatility, and the inference is that the heavier elements are more deeply seated in the body of the sun.

Or, again, when the nebula from which our system was formed was contracting and breaking up into sun and planets, is there any reason why that small fraction which became our earth should necessarily have been a fair sample of the whole nebula? Or, indeed, need we suppose that the elements were originally formed in equal proportions?—I am, &c.,

J. C. KINGDON.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxvii., No. 25, December 21, 1903.

### PRIZE NUMBER.

The Jecker Prize is awarded to L. BOUVEAULT for his researches in organic chemistry. His chief researches during later years were on the condensation of aldehydes with ordinary acetone leading to the synthesis of unsaturated ketones. He has also investigated the complex question of camphor and the terpenes and prepared a new liquid hydrocamphene, besides investigating the constitution of compounds of the isolaric and  $\beta$ -campholenic series.

The La Caze Prize is given to M. GUNTZ for his various researches on general chemistry. His later researches have been connected with the electrolytic preparation of lithium, by which method he has prepared large quantities of the metal, and so been enabled to investigate its properties more fully.

No. 26, December 28, 1903.

Density of Chlorine.—H. Moissan and Binet du Jassoneix.—The authors find that if the density of chlorine is taken by Dumas' method, when the chlorine is prepared under ordinary conditions, the density varies between 2.424 and 2.506. The principal causes of error in these determinations are (1) the presence of air in the density bulb and in the current of chlorine, led through the



**Contribution to the Study of Cuprous Compounds.**—G. Bodlander and O. Storbeck.—The examination of the solubility of cuprous bromide shows the decomposition of the salt into cupric bromide and copper. In the double salts formed by cuprous bromide with the other metallic bromides, KBr for example, the complex ions appear to have the formula  $(\text{CuBr}_2)_n^{+}$  for fairly strong concentrations of the metallic chloride, while with slight concentrations these ions correspond to  $(\text{CuBr}_2)^+$ . *The Cuprous Ions are Monovalent.*—The potential of discharge of the cuprous

ions is  $-0.454$  volt, that of the cupric ions  $-0.329$ , ( $H=0$ ). The equilibrium between the cupric ions and iodine can be expressed by the formula  $2Cu^{++} + 2I^- = 2Cu^+ + I_2$ .

|            | Product of solubility. | Energy of formation. | Heat of formation. |
|------------|------------------------|----------------------|--------------------|
| CuCl .. .. | $1.20-10^{-6}$         | 30'000               | 32'900             |
| CuBr .. .. | $4.15-10^{-8}$         | 22'300               | 25'000             |
| CuI .. ..  | $5.06-10^{-12}$        | 16'600               | 16'300             |

—*Zeit. Anorg. Ch.*, vol. xxxi., p. 458.

**Simultaneous Volumetric Estimation of Boric Acid and Strong Acids.**—W. Herz.—The principle of this method consists in titrating the strong acid by using an indicator on which the boric acid is without action. An estimation of the boric acid is then effected, after the addition of alcohol, mannite, or glycerin, by titrating in the usual manner with phenolphthalein. The indicator used for the titration of the strong acid is nitrophenol; the colourless solution becomes yellow in the presence of an excess of alkali. The following figures show what results may be expected by this method:—

Composition of the mixture—

|                                  |      |      |       |      |       |       |
|----------------------------------|------|------|-------|------|-------|-------|
| HCl ..                           | 13'0 | 6'5  | 19'5  | 6'5  | 26'0  | 21'7  |
| B <sub>2</sub> O <sub>3</sub> .. | 7'78 | 3'89 | 1'945 | 5'83 | 1'945 | 1'945 |
| Found—                           |      |      |       |      |       |       |
| HCl ..                           | 13'0 | 6'5  | 19'4  | 6'45 | 26'0  | 21'75 |
| B <sub>2</sub> O <sub>3</sub> .. | 7'78 | 3'99 | 1'99  | 5'73 | 1'99  | 1'945 |

—*Zeit. Anorg. Ch.*, vol. xxxiii., p. 353.

**Chemical Study of the Culture of Chrysanthemums.**—A. Hébert and G. Truffaut.—The authors find that three elements are necessary: nitrogen, phosphoric acid, and potash. Chrysanthemums are particularly greedy for phosphoric acid; this substance acts particularly on the formation of chlorophyll. Potash also plays an important part; it provokes turgescence of all the organs. Finally, nitrogen is an indispensable element; its absence causes the formation of rachitic and chlorotic plants. An excess of nitrogen is frequently the cause of cryptogamic maladies. —*Bull. Soc. Chim.*, Series 3, xxix., No. 12.

## MEETINGS FOR THE WEEK.

- MONDAY, Feb. 1st.**—Society of Arts, 8. (Cantor Lectures). "Oil and Fate—their Uses and Applications," by J. Lewkowitch, Ph.D., &c.  
Royal Institution, 5. General Monthly Meeting.
- TUESDAY, 2nd.**—Royal Institution, 5. "Development of Animals," by Prof. L. C. Miall, F.R.S.  
Faraday Society, 8. "Notes on Aluminium Welding," by Sherard Cowper-Coles. "Some Applications of the Theory of Electrolysis to the Separation of Metals from one another," by A. Holland.
- WEDNESDAY, 3rd.**—Society of Arts, 8. "Steam Cars for Public Service," by Thomas Clarkson.  
Society of Public Analysts, 8. (Annual Meeting). President's Address. "Note on the Quantitative Estimation of Mechanical Wood Pulp in Paper," by C. F. Cross and E. J. Bevan. "Note on Chinese Tallow Seed Oil," by L. Myddelton Nash. "Note on the Analysis of Jam," by Raymond Ross.
- THURSDAY, 4th.**—Royal Institution, 5. "Recent Research in Agriculture," by A. D. Hall, M.A.  
Chemical, 8. "The Tautomeric Character of the Acidic Thiocyanates," by R. E. Doran. "The Resolution of  $\alpha,\beta$ -Dihydroxybutyric Acid into its Optically Active Constituents," by R. S. Morrell and E. K. Hanson.
- FRIDAY, 5th.**—Royal Institution, 9. "The Growing Distaste for the Higher Kinds of Poetry," by Alfred Austen, B.A.
- SATURDAY, 6th.**—Royal Institution, 3. "The Study of Style in Greek Sculpture," by Charles Walostein, Litt.D.

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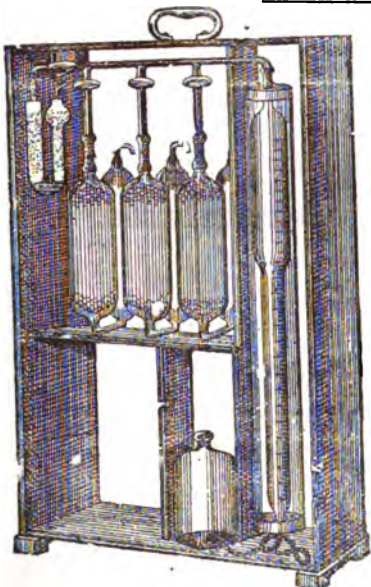
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### CONTENTS.

| ARTICLES:—                                                                                                                                                                                                                                                                                                                                 | PAGE |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Estimation of Unburnt Products in Chimney Gases by means of a Modified Orsat Apparatus, by William H. Sodean, B.Sc., F.I.C. ....                                                                                                                                                                                                       | 61   |
| Precipitation and Separation by Weak Organic Bases, by E. T. Allen.....                                                                                                                                                                                                                                                                    | 63   |
| The Position of Chemistry in the Medical Curriculum of the University of London.....                                                                                                                                                                                                                                                       | 65   |
| PROCEEDINGS OF SOCIETIES—                                                                                                                                                                                                                                                                                                                  |      |
| CHEMICAL SOCIETY.—Report of the International Committee on Atomic Weights—Chemical Reactions of Nickel Carbonyl: (1) Reactions with the Halogens, &c.; (2) Reaction with Aromatic Hydrocarbons in presence of Aluminium Chloride, Synthesis of Aldehydes and Anthracene Derivatives—Optically Active Asymmetric Nitrogen Compounds—&c..... | 67   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                                                                                                                                                                                                                | 70   |
| MISCELLANEOUS .....                                                                                                                                                                                                                                                                                                                        | 71   |
| NOTES AND QUERIES.....                                                                                                                                                                                                                                                                                                                     | 72   |
| MEETINGS FOR THE WEEK.....                                                                                                                                                                                                                                                                                                                 | 72   |

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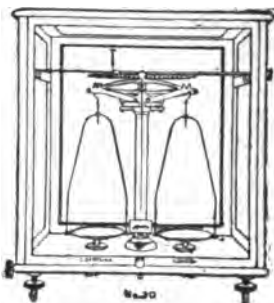
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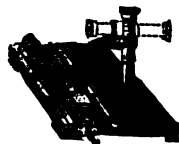
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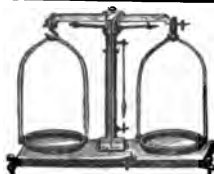
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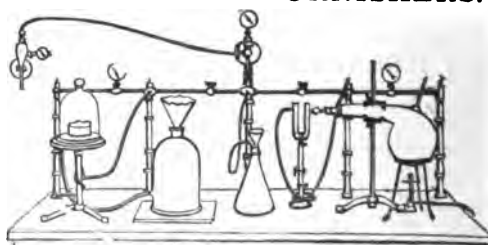
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2306.

## THE ESTIMATION OF UNBURNT PRODUCTS IN CHIMNEY GASES BY MEANS OF A MODIFIED ORSAT APPARATUS.

By WILLIAM H. SODEAU, B.Sc., F.I.C.  
Elswick Works and Walker Shipyard, Newcastle-on-Tyne.

In experimental work on the economic application of fuel, it is very desirable to know, whilst a trial is actually proceeding, not only what excess of air is being employed, but also the amount of unburnt gases.\* Combining these data with the indications of a thermojunction placed at the base of the funnel, one can follow throughout the trial the total amount of heat (potential as well as actual) which is passing up the chimney. With a given boiler, for example, if one takes care of the exit gases, the evaporation will practically take care of itself. The fuel employed represents a certain total amount of heat, and the loss by radiation from the boiler will be practically uniform; hence the evaporation per pound of fuel may be gauged by what remains after deducting the various losses from the calorific value of the fuel, or, in other words, by working up the chimney gas results as in a "heat balance sheet."

This method of checking the evaporative efficiency is especially advantageous in short experimental runs, as feed water is not always read with much accuracy, and all that passes through the main stop valve may not actually be steam.

The actual analytical problem may be reduced to the rapid determination of carbon dioxide, carbon monoxide, and hydrogen (including hydrocarbons if present). It is, however, customary to determine the oxygen in chimney gases, whilst the estimation of hydrogen is usually omitted. The desirability of determining the amount of hydrogen is illustrated by the table below, in which are given a few analyses of the products of incomplete combustion obtained in some experiments with a 1000 horse-power water-tube boiler of the "Express" type. With Welsh coal the loss as unburnt hydrogen usually amounted to nearly a third of the loss as carbon monoxide, whilst with petroleum the proportion averaged about two-thirds. This difference was no doubt mainly due to the larger proportion of total hydrogen present when oil-fuel was employed.

Air being practically constant in composition it is unnecessary to estimate the oxygen in chimney gases if the approximate composition of the fuel is known, for it is then easy to work out simple formulæ by means of which those data which are of practical importance may be calculated with fair accuracy from the percentages of carbon dioxide, carbon monoxide, and hydrogen alone.

\* When working with very limited furnace space, as in naval water-tube boilers of the "Express" type, a reduction of the "excess" of air may cause large amounts of combustible gases to escape unburnt, and so lead to decreased, instead of increased, evaporative efficiency.

Take, for example, two analyses of fuels:—

|                       | Welsh coal. | Texas petroleum. |
|-----------------------|-------------|------------------|
| Carbon .. .. .        | 88.2        | 85.0             |
| Sulphur .. .. .       | 0.77        | 1.34             |
| Hydrogen .. .. .      | 2.6         | 12.0             |
| Ash, oxygen, &c. .. . | 8.43        | 1.66             |

|                                                 |           |            |
|-------------------------------------------------|-----------|------------|
| Theoretical amount of air for 1 pound fuel .. . | 11.1 lbs. | 13.95 lbs. |
|-------------------------------------------------|-----------|------------|

Using CO<sub>2</sub>, CO, and H<sub>2</sub> to respectively denote the percentages of carbon dioxide, carbon monoxide, and hydrogen in the chimney gases, the following formulæ may be obtained by calculation from the equations for combustion:—

1. Pounds air per pound of fuel—  

$$\frac{204}{\text{CO}_2 + \text{CO}} + 0.28$$

$$\frac{206}{\text{CO}_2 + \text{CO}} + 0.84$$
2. Excess air per cent above theoretical—  

$$\frac{1837 - 9 \text{ CO}}{\text{CO}_2 + \text{CO}} - 97.5$$

$$\frac{1476 - 7.4 \text{ CO}}{\text{CO}_2 + \text{CO}} - 94.1$$
3. Evaporation units lost as unburnt gases—  

$$9.33 \frac{\text{CO} + \text{H}_2}{\text{CO}_2 + \text{CO}}$$

$$9.04 \frac{\text{CO} + \text{H}_2}{\text{CO}_2 + \text{CO}}$$

These formulæ will, of course, apply only to fuels having the composition given above. For ordinary purposes the terms including CO may be omitted from the numerators of formulæ (1) and (2). Curves can then be plotted having CO<sub>2</sub>+CO on one axis, and either "pounds air per lb. of fuel" or "excess air per cent above theoretical" on the other, so that the meaning of an analysis can be seen at a glance.

An ordinary Orsat apparatus affords a ready means of estimating the carbon dioxide, but the absorption of carbon monoxide by means of that somewhat objectionable reagent, cuprous chloride, involves the previous removal of oxygen by means of phosphorus or alkaline pyrogallol, absorbents which act exceedingly slowly when too cold. This method takes no account of free hydrogen (or saturated hydrocarbons). The author decided to discard absorption by cuprous chloride in favour of a combustion method, and not finding the capillary combustion tube of palladinised asbestos (as fitted in the Orsat-Lunge apparatus) quite suitable for use in the stokehold, finally adopted an Orsat apparatus modified as shown in the accompanying figure, in which the main feature is an adaptation of the Winkler combustion pipette.\*

A large glass stopcock, s, of 4 to 5 m.m. clear bore, is attached to the base of the apparatus, one end being connected to the measuring tube and the other to the reservoir, m r. The pipette, k, is of the ordinary form, and contains caustic potash solution (one part potash to two of water). A similar pipette, p, containing phosphorus or alkaline

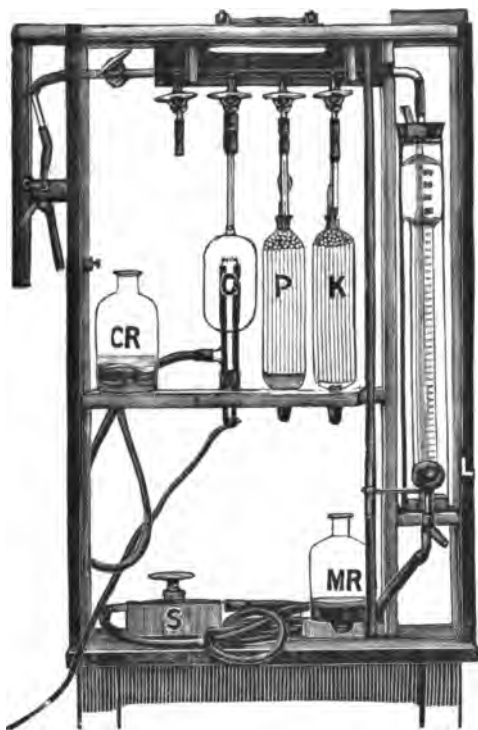
\* The Winkler pipette, a development of Coquillon's "Grisoumeter," is described and figured in Winkler and Lunge's "Technical Gas Analysis," pp. 151—155.

### Examples of Incomplete Combustion.

|                                                                        | Welsh coal. |      |       |      | Crude Texas oil (steam sprayed). |       |       |      |
|------------------------------------------------------------------------|-------------|------|-------|------|----------------------------------|-------|-------|------|
| Carbon dioxide, per cent .. .. .                                       | 9.0         | 11.0 | 9.9   | 9.2  | 6.1                              | 9.5   | 9.0   | 7.9  |
| Carbon monoxide, per cent .. .. .                                      | 2.15        | 2.8  | 1.65  | 1.3  | 2.1                              | 1.5   | 1.0   | 0.6  |
| Hydrogen, per cent .. .. .                                             | 0.65        | 0.55 | 0.47  | 0.4  | 1.2                              | 1.0   | 0.6   | 0.4  |
| Pounds air per pound fuel .. .. .                                      | 17.2        | 14.7 | 17.75 | 19.5 | 25.7                             | 19.45 | 21.35 | 25.0 |
| Excess air per cent above theoretical .. .                             | 55.0        | 32.3 | 60.0  | 75.7 | 84                               | 39    | 53    | 79   |
| Evaporation units* per pound of fuel lost as combustible gases .. .. . | 2.34        | 2.26 | 1.71  | 1.51 | 3.65                             | 2.05  | 1.45  | 1.06 |

\* An "evaporation unit" is the amount of heat required to convert one pound of water at 100° C. into steam at the same temperature.

pyrogallol, may be used if it is desired to estimate the oxygen. The combustion pipette, c, may be made from the commercial form of Winkler pipette by simply cutting off the U-tube and sealing on a straight piece of capillary tube of suitable length. An ordinary Hempel pipette for solid absorbents may be altered in a similar manner, the neck at the bottom being closed by a two-holed indiarubber stopper through which passes a pair of unlacquered brass electrodes bridged across by a platinum spiral made by coiling about 4 c.m. of platinum wire of about 0.3 m.m. diameter around a needle 1.3 m.m. thick.



When the combustion pipette is employed with mixtures rich in combustible gases the coil must be near the top of the bulb in order that serious explosions may be avoided, but for the analysis of any ordinary chimney gases the coil may be placed much lower in order to reduce the heating of the glass. The gas may then be returned as soon as the current is cut off, without waiting for the glass to cool.

A fixed bulb\* as in the Hempel (or Orsat) pipette, may be employed to receive the displaced water unless it is desired to carry out the rapid estimation of carbon monoxide and hydrogen together, as described below, when the bulb, c, should be connected by means of indiarubber tubing to the reservoir, c r, consisting of a small aspirator bottle, similar to that connected to the measuring tube. When in use the spiral is raised to a white heat by means of a two-cell accumulator, the current being conveniently adjusted to the right strength by passing it through a few feet of the tinned iron wire, about  $\frac{1}{4}$ " diameter, which is commonly sold in penny skeins.

In order to eliminate parallax the measuring tube is read by means of a lens mounted in conjunction with an eyecap, and sliding on a brass rod, as employed in connection with the "Kew principle" correction tube (*Journ. Soc. Chem. Ind.*, 1903, xxii., pp. 188, 189). Before each reading

\* The ordinary Hempel pipette is sometimes supplied with a bulb too small to contain the water displaced by the heated gas.

is taken, the water in the measuring tube is allowed to drain down for one minute, as indicated by the sand-glass used for timing the absorptions and combustions.

The U-tube filled with glass wool ordinarily supplied with the Orsat apparatus being somewhat apt to clog if dense black smoke is produced, it is conveniently replaced by a simple T piece having a small plug of glass wool in the limb through which the sample enters the apparatus. In this way only the actual samples are filtered, and the solid particles in the main stream pass direct to the aspirator.

**Method of Analysis.**—The measuring off of the sample is effected in the usual manner, except that it is convenient to close s instead of pinching the indiarubber tube when adjusting the water to the zero mark prior to bringing the gas to atmospheric pressure.

Carbon dioxide is estimated by sending the greater part of the gas over into k and back again, then leaving it for one minute to complete the absorption, and measuring again as usual; the decrease gives the percentage of carbon dioxide. The current is next switched on, and the gas passed over into the combustion pipette, c, where it remains for one minute, being then returned to the measuring tube (after switching off the current), and the contraction noted.

The carbon dioxide produced is then determined by a one minute absorption in the potash pipette, k; the decrease gives the percentage of carbon monoxide.

As carbon monoxide unites with half its volume of oxygen to form its own volume of carbon dioxide, it follows that half the amount of the carbon monoxide found must be deducted from the contraction during combustion. Two-thirds of the corrected contraction equals the percentage of hydrogen.

For example, if the contraction on combustion amounted to 2.3 c.c. and the resulting carbon dioxide to 1.6 c.c., then the gas contained 1.6 per cent of carbon monoxide and  $\frac{2}{3}(2.3 - \frac{1.6}{2}) = \frac{2}{3} \times 1.5 = 1.0$  per cent of hydrogen. It

should be noted that the only absorbent employed is one which is readily obtained and seldom needs renewing. The estimation of carbon dioxide, carbon monoxide, and hydrogen by the above method can be completed in fifteen minutes. Any traces of hydrocarbons, if present, will of course appear partly as carbon monoxide and partly as hydrogen in the above method of analysis. Their heat value will not be fully represented, but this is an unimportant defect, and it exists more markedly in the old cuprous chloride method.

It may occasionally be desirable to know the percentage of oxygen originally present in the gas. This may be found by finally absorbing with phosphorus or pyro in the pipette, p, and adding to the result the amount of oxygen required to burn the combustible gases.

**Rapid Joint Estimation of Carbon Monoxide and Hydrogen.**—This may be carried out by omitting the measurement immediately after combustion, and transferring the gas direct from c to k in the following manner:—After the carbon dioxide has been determined, open the stopcock attached to c, and switch on the current, raising the reservoir, m r, so as to drive the gas into c. Close s when the water has risen to the top of the measuring tube, and about one minute later switch off the current and open the stopcock of k, raising the reservoir, c r, so as to drive the gas over into the potash, and finally closing the stopcock of c. After allowing one minute for absorption, the stopcock, s, is opened, the residue drawn back into the measuring tube, and the stopcock of k then closed. Two-thirds of the contraction so produced equals the percentage of "combustible gases," i.e., carbon monoxide and hydrogen, in the sample.

The result so obtained is translated into "evaporation units" by means of a formula similar to 3, the denominator being replaced by  $\text{CO}_2 + (\frac{2}{3} \times \text{combustible gases})$  in the case of Welsh coal, and by  $\text{CO}_2 + (\frac{1}{3} \times \text{combustible gases})$  when



Texas oil is employed. The amount of air is similarly found by means of formulæ or curves.

When collecting samples of chimney gas for subsequent analysis the sometimes troublesome process of saturating water during the trial may be avoided by appropriately diluting a saturated solution of carbon dioxide. Thus, if the gas is expected to contain about 12 per cent of carbon dioxide, the sample bottles should be filled with a mixture of seven parts tap-water and one part of water saturated with carbon dioxide. The flow of water from the sample bottle may be conveniently regulated by attaching, say, a yard of tubing (in order to give a fairly uniform head), to the end of which is connected a "wash-bottle jet" which has previously been found to permit the emptying to take place in the required time. The jet is less likely to clog if used in the reversed position.

## PRECIPITATION AND SEPARATION BY WEAK ORGANIC BASES.\*

By E. T. ALLEN.

(Continued from p. 44).

### Separation of small quantities of Alumina from excess of Iron.

The first precipitation in the following series was made in a nearly neutral solution of about 150 c.c. volume. After washing out most of the iron by the phenylhydrazine sulphite solution, the precipitate was dissolved on the filter in a few cubic centimetres of hot dilute hydrochloric acid, washed through with hot water, neutralised with ammonia, and acidulated with 2 or 3 drops 1:1 HCl. The precipitate which is thrown down in a small volume of liquid by 0.5 c.c. phenylhydrazine, is washed with the sulphite solution till free from iron. After blasting, the precipitate is usually pure white.

| No. | FeCl <sub>3</sub> taken.<br>C.c. | Fe <sub>2</sub> O <sub>3</sub> .<br>Grm. | AlCl <sub>3</sub> taken.<br>C.c. | Al <sub>2</sub> O <sub>3</sub> .<br>Grm. | Al <sub>2</sub> O <sub>3</sub> found.<br>Grm. | Error.<br>Grm. |
|-----|----------------------------------|------------------------------------------|----------------------------------|------------------------------------------|-----------------------------------------------|----------------|
| 1.  | 35                               | = 0.2189                                 | 0.1                              | = 0.0005                                 | 0.0005                                        | 0.0000         |
| 2.  | 35                               | = 0.2189                                 | 0.2                              | = 0.0010                                 | 0.0014                                        | +0.0004        |
| 3.  | 35                               | = 0.2189                                 | 0.6                              | = 0.0030                                 | 0.0031                                        | +0.0001        |
| 1'. | 35                               | = 0.2189                                 | 0.7                              | = 0.0007                                 | 0.0008                                        | +0.0001        |
| 2'. | 35                               | = 0.2189                                 | 1.2                              | = 0.0012                                 | 0.0013                                        | +0.0001        |
| 3'. | 33                               | = 0.1867                                 | 2.0                              | = 0.0020                                 | 0.0019                                        | -0.0001        |

### Separation of Titanium from Iron.

As solutions of titanium chloride are quite unstable, a standard solution of the sulphate was prepared as follows:—Potassium titanio-fluoride of the market was twice crystallised from hot water and dried at 105° C. A weighed quantity of this compound was then heated in a large platinum crucible with excess of sulphuric acid until free from fluorine. A very low flame should be used, or insoluble basic sulphate may form. The cooled sulphate was then poured into excess of cold water, precipitated by ammonia, washed, and re-dissolved in a quantity of sulphuric acid 5 or 10 per cent in excess of that demanded by theory, to prevent hydrolysis.

Phenylhydrazine gives a practically complete separation of titanium from iron in two precipitations. I have never found more than traces of titanium remaining with the iron. It was sought for as follows:—The iron, with any titanium it might contain after the precipitation with phenylhydrazine, was removed from the solution by ammonium sulphide, washed a few times, and dissolved in nitric acid, from which solution it was subsequently precipitated by ammonia, and weighed. The weighed oxide was now tested for titanium by the method described in *Bulletin* 176, U.S. Geol. Survey, p. 67.

|                                                          |         |              |
|----------------------------------------------------------|---------|--------------|
| TiO <sub>2</sub> found in Fe <sub>2</sub> O <sub>3</sub> | I. ..   | 0.00017 grm. |
|                                                          | II. ..  | none         |
|                                                          | III. .. | 0.0002       |
|                                                          | IV. ..  | 0.0002       |
|                                                          | V. ..   | trace        |

The same details apply to this separation as were laid down for the separation of aluminum from iron, except that here the solution may be considerably more acid before adding the phenylhydrazine. If much titanium is present, a considerable precipitate will form before the solution is completely neutralised by ammonia. When the neutral-point is reached, a half-dozen drops of 1:1 hydrochloric acid may be added. This quantity is frequently insufficient to re-dissolve all the titanium precipitate, but that is a matter of no consequence. (See Table A).

### Separation of Zirconium from Iron.

A standard solution of zirconium sulphate was used in these experiments. The raw material consisted of picked crystals of North Carolina zircon,\* which were converted into potassium zircono-fluoride after the excellent method of Marignac (*Ann. Chim., Phys.*, [3], lx., 260).

After two crystallisations it was dried carefully at 105° C., and then changed into sulphate by a method similar in all details to that given for the titanium compound. 1.0470 grm. of the above preparation, thus treated and afterwards precipitated by ammonia, thoroughly washed, dissolved in hydrochloric acid, and again precipitated and washed as before, ignited, and finally blasted, gave 0.4508 grm. ZrO<sub>2</sub> instead of 0.4529 grm. required for K<sub>2</sub>ZrF<sub>6</sub>.

The details of the separation of zirconium from iron are the same as those for the separation of titanium. The results given in Table B. show that a single precipitation is not always sufficient to remove all the iron.

### Separation of Aluminum, Titanium, and Zirconium from Iron.

In the series of determinations, given in Table C., aluminum, zirconium, and titanium in varying quantities were separated from iron as already described, and weighed together. The standard solutions used have already been described.

In a recent paper by Miss A. M. Jefferson (*Journ. Am. Chem. Soc.*, xxiv., 543) it is stated that zirconium is not precipitated by phenylhydrazine. Now zirconia is certainly a weaker base than alumina, and, according to the principles stated in the beginning of this paper, ought to be precipitated by phenylhydrazine, the more so since, according to Miss Jefferson, it is precipitated by aniline, a base which my experimentis indicate to be somewhat weaker than phenylhydrazine. It should be noted, however, that Miss Jefferson employed a cold solution of zirconium nitrate, while the experiments I have described were performed with solutions containing sulphates and chlorides, or chlorides alone. As theory indicates the possibility of a difference, the following tests were undertaken:—Zirconium hydroxide was prepared from the sulphate, and then dissolved in dilute nitric acid. The solution was freed from most of the acid by evaporating to a volume of a few c.c., and diluted with cold water so that 1 c.c. contained about 1 m.grm. ZrO<sub>2</sub>. The solution contained no sulphate. In the first test, a portion of it was precipitated by a large excess of phenylhydrazine, 5 c.c. to 25 c.c. of the solution. After standing more than twenty-four hours, the precipitate was filtered, washed, ignited, and blasted.

|                    |        |             |
|--------------------|--------|-------------|
|                    | Found. | By ammonia. |
| ZrO <sub>2</sub> = | 0.0251 | 0.0245      |

Twenty-five c.c. of the solution with 1 c.c. of the reagent gave ZrO<sub>2</sub> = 0.0241 grm. Ammonia gave no precipitate with the filtrate. I could not discover that the zirconium

\* From the *Journal of the American Chemical Society*, xxv., No. 4.

\* My thanks are due to Mr. Wirt Tassin, Assistant Curator of the National Museum, from whom the zircon, as well as a supply of beryl, was obtained.

TABLE A.

| No. | FeCl <sub>3</sub> taken.<br>C.c. | Fe <sub>2</sub> O <sub>3</sub> .<br>Grm. | Fe <sub>2</sub> O <sub>3</sub> found.<br>Grm. | Error.<br>Grm. | Ti(SO <sub>4</sub> ) <sub>2</sub> taken.<br>C.c. | TiO <sub>2</sub> .<br>Grm. | TiO <sub>2</sub> found.<br>Grm. | Error.<br>Grm. |
|-----|----------------------------------|------------------------------------------|-----------------------------------------------|----------------|--------------------------------------------------|----------------------------|---------------------------------|----------------|
| 1.  | 35                               | =                                        | 0.1748                                        | —              | 2                                                | =                          | 0.0020                          | +0.0002        |
| 2.  | 5                                | =                                        | 0.0250                                        | +0.0004        | 25                                               | =                          | 0.0246                          | -0.0009        |
| 3.  | 2                                | =                                        | 0.0100                                        | +0.0004        | 25                                               | =                          | 0.0246                          | +0.0008        |
| 4.  | 25                               | =                                        | 0.1248                                        | —              | 1                                                | =                          | 0.0010                          | +0.0001        |
| 5.  | 10                               | =                                        | 0.0499                                        | —              | 50                                               | =                          | 0.0492                          | +0.0009        |
| 6.  | 3                                | =                                        | 0.0140                                        | —              | 50                                               | =                          | 0.0492                          | +0.0010        |

TABLE B.

| No.                   | FeCl <sub>3</sub> taken.<br>C.c. | Fe <sub>2</sub> O <sub>3</sub> .<br>Grm. | Zr(SO <sub>4</sub> ) <sub>2</sub> taken.<br>C.c. | TiO <sub>2</sub> .<br>Grm. | ZrO <sub>2</sub> found.<br>Grm. | Error.<br>Grm. |
|-----------------------|----------------------------------|------------------------------------------|--------------------------------------------------|----------------------------|---------------------------------|----------------|
| Single Precipitation. |                                  |                                          |                                                  |                            |                                 |                |
| 1.                    | 30                               | =                                        | 0.1876                                           | 2                          | =                               | +0.0003        |
| 2.                    | 4                                | =                                        | 0.0250                                           | 23                         | =                               | +0.0001        |
| 3.                    | 1                                | =                                        | 0.0062                                           | 25                         | =                               | +0.0006        |
| 4.                    | 35                               | =                                        | 0.2189                                           | 1                          | =                               | +0.0006        |
| 5.                    | 10                               | =                                        | 0.0625                                           | 50                         | =                               | +0.0022        |
| 6.                    | 1                                | =                                        | 0.0062                                           | 50                         | =                               | +0.0032        |
| Double Precipitation. |                                  |                                          |                                                  |                            |                                 |                |
| 1.                    | 50                               | =                                        | 0.2997                                           | 50                         | =                               | -0.0004        |
| 2.                    | 50                               | =                                        | 0.2997                                           | 1                          | =                               | +0.0001        |
| 3.                    | 5                                | =                                        | 0.0300                                           | 25                         | =                               | -0.0002        |

TABLE C.

| No.        | Fe <sub>2</sub> O <sub>3</sub> taken. | Al <sub>2</sub> O <sub>3</sub> taken. | TiO <sub>2</sub> taken. | ZrO <sub>2</sub> taken. | Al <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub> + ZrO <sub>2</sub> |                | Error.    |
|------------|---------------------------------------|---------------------------------------|-------------------------|-------------------------|----------------------------------------------------------------------|----------------|-----------|
|            | Grm.                                  | Grm.                                  | Grm.                    | Grm.                    | taken.<br>Grm.                                                       | found.<br>Grm. |           |
| 1.         | 0.0999                                | 0.1500                                | 0.00985                 | 0.00492                 | 0.1648                                                               | 0.1654         | + 0.0006  |
| 2.         | 0.1249                                | 0.1650                                | 0.01477                 | 0.00196                 | 0.1817                                                               | 0.1816         | - 0.0001  |
| 3.         | 0.1748                                | 0.0500                                | 0.0197                  | 0.00287                 | 0.0726                                                               | 0.0717         | - 0.0009  |
| 4.         | 0.0749                                | 0.1000                                | 0.0049                  | 0.00098                 | 0.1059                                                               | 0.1063         | + 0.0004  |
| 5.         | 0.1748                                | 0.0250                                | 0.0197                  | 0.0148                  | 0.0595                                                               | 0.0603         | + 0.0008  |
| Average .. |                                       |                                       |                         |                         | ..                                                                   | ..             | + 0.00016 |

hydroxide had any tendency to dissolve in excess of phenylhydrazine. The same statement may be made of aniline, which also precipitates zirconium completely.

It was thought to be of some interest to determine the behaviour of phenylhydrazine with the remaining elements of this natural family, thorium and cerium.

#### Phenylhydrazine and Thorium.

A solution of thorium nitrate was prepared from the pure dioxide. The latter was brought into solution by fusion with potassium acid sulphate, from which it was precipitated by ammonia in a form soluble in dilute nitric acid. A double precipitation was necessary to remove sulphate entirely. The nitrate was evaporated to crystallisation, and dissolved in cold water.

Three portions of 25 c.c. each were then precipitated as follows:—No. 1 was precipitated by ammonia, washed, ignited, and weighed as in an ordinary determination. No. 2 was nearly neutralised with ammonia, precipitated hot, and washed with hot water. No. 3 was treated as No. 2, except that a smaller amount of reagent was added to the cold solution, and the precipitate was washed with cold water. In all cases the precipitate was blasted before weighing.

#### ThO<sub>2</sub> found.

1. 25 c.c. contained 0.0477 grm. average of three determinations by NH<sub>3</sub>.
2. 25 c.c. hot by phenylhydrazine 0.0477 grm.
3. 25 c.c. cold by phenylhydrazine 0.0475.

The precipitated thorium was white or nearly so.

Regarding Miss Jefferson's statement (*Yourn. Am. Chem. Soc.*, xxiv., 546) that phenylhydrazine throws down, from a solution of thorium nitrate, "a canary-yellow precipitate,

easily soluble in excess," I can say that if the solution contains much free nitric acid, the precipitate is coloured yellow, evidently by an oxidation product of the phenylhydrazine. The precipitate may be obscured by an excess of reagent, but there is no tendency to re-dissolve. Thus, in one test I employed 5 c.c. of the reagent to a small volume of the thorium solution. I was at first in doubt about the result, but a careful examination showed that no thorium passed through an ordinary filter.

#### Phenylhydrazine and Cerium.

The remaining element in the family under consideration is cerium. It differs from the other three members, in that it is easily reducible from the quadrivalent to the trivalent state. Phenylhydrazine fails to precipitate it except in a partial way from its ceric salts, not because it is a more basic element than the rest, but because it is an oxidising agent which phenylhydrazine easily reduces to the cerous condition, and cerous oxide is a much stronger base. These statements were proved by the following experiments:—

Ceric ammonium nitrate, (NH<sub>4</sub>)<sub>2</sub>Ce(NO<sub>3</sub>)<sub>6</sub>.14H<sub>2</sub>O, was made from a preparation of cerous nitrate furnished by Merck. To separate it from any neodymium and praseodymium, it was twice precipitated by caustic potash and oxidised by a stream of chlorine, the ceric hydroxide being thoroughly washed after each treatment. The ceric hydroxide was now changed to ceric ammonium nitrate by the addition of dilute nitric acid and a little ammonium nitrate. A comparatively concentrated solution of this salt, containing considerable free acid, at once oxidised both phenylhydrazine and aniline with marked colour changes, and no excess of either produced, thereafter, any precipitate (see also "Gmelin-Kraut," vol. ii., Part I., p.

508). With a dilute and nearly neutral solution, the results were different. Such a solution was obtained by evaporating one like the above to dryness in a vacuum desiccator with lime and sulphuric acid.

The beautiful orange crystals, with the small excess of ammonium nitrate they contained, were then put into solution by cold water, and after standing a day or two, filtered from a slight precipitate which had formed. The concentration of this solution was not far from 1 m.grm.  $\text{CeO}_2$  per c.c. (20 c.c. gave 0.0161 grm.). Fifteen c.c. of the solution were treated with a little phenylhydrazine and set away for twenty-four hours. Some little precipitate, composed partly of organic matter, apparently an oxidation product of the reagent, had formed, but the filtrate yielded with ammonia 0.0078 grm.  $\text{CeO}_2$  out of a total quantity of 0.0121 grm. originally contained in the solution. Aniline being a weaker reducing agent gave a practically complete precipitation with this solution. Only two or three very small flakes were obtained from the filtrate by ammonia.

#### Phenylhydrazine and Ferric Salts.

Ferric salts follow ceric salts in their behaviour, except that phenylhydrazine gives with the former almost no precipitate. This is true, at least, with both strong and weak solutions of ferric chloride. With one containing about 1 m.grm. per c.c., phenylhydrazine at first brings down some brown hydroxide, which dissolves when the liquid is shaken, leaving only a little insoluble tarry matter. Aniline, however, gives a complete precipitation; at least the filtrate is colourless, and gives no precipitate with ammonia.

(To be continued).

### THE POSITION OF CHEMISTRY IN THE MEDICAL CURRICULUM OF THE UNIVERSITY OF LONDON.

#### A.—RESOLUTIONS ADOPTED BY THE RECOGNISED TEACHERS OF CHEMISTRY IN MEDICAL SCHOOLS OF LONDON, DECEMBER 5TH, 1903.

(Submitted to the Academic Council of the University).

In view of the changes in the character of the requirements relating to the teaching of chemistry for medical degrees of the London University, at a meeting of the recognised teachers of chemistry at medical schools the following resolutions were unanimously passed:—

1. That instruction in physiological and pathological chemistry can in no sense be regarded as taking the place of instruction in the pure scientific organic chemistry hitherto required by the University.
2. That this instruction in organic chemistry can only properly be given in a chemical laboratory and by a chemist.
3. That a careful scientific study of organic chemistry is essential for any later intelligent study of physiological and pathological chemistry.
4. That this study of organic chemistry must be preceded by a study of inorganic chemistry.
5. That to properly carry out such an education the Preliminary Scientific Examination in chemistry might be made identical with the present Intermediate Science Examination for internal students; and that to provide for the proper study of organic chemistry necessary a further period is required.
6. That to relieve the student the examination should be in two parts:—(1) Inorganic chemistry identical with the present Intermediate Science Examination for internal students, and (2) an examination in organic chemistry, held at convenient times.
7. That in the opinion of the teachers of chemistry it would be possible for a student to take the examination in both inorganic and organic chemistry at

the end of one academic year, only when he has taken chemistry at the Matriculation Examination.

8. That it is desirable that a more detailed syllabus in chemistry for the Preliminary Scientific Examination for internal students be prepared.

In the event of the University favouring the above suggestions, the teachers of chemistry in the medical schools of the University would be prepared to submit a schedule for consideration by the Board of Studies in Chemistry.

These resolutions were supported by the University Professor of Chemistry, and the Lecturers on Chemistry in the Medical Schools of the University:—

Hugh Candy, B.A., B.Sc.; F. D. Chattaway, M.A., D.Sc., Ph.D.; Arthur W. Crossley, D.Sc.; Clare de B. Evans, D.Sc.; J. Addyman Gardner, M.A.; H. Wilson Hake, Ph.D.; A. M. Kellas, B.Sc., Ph.D.; H. Forster Morley, M.A., D.Sc.; Frederic J. M. Page, B.Sc.; Sir W. Ramsay, Sc.D., F.R.S.; John M. Thomson, LL.D., F.R.S.; John Wade, D.Sc.; W. H. Willcox, M.D., B.Sc.

#### B.—RECOMMENDATIONS OF THE RECOGNISED TEACHERS OF CHEMISTRY IN MEDICAL SCHOOLS OF LONDON.

(Drawn up at the Request of the Vice-Chancellor of the University).

December 22nd, 1903.

In view of the changes in the character of the regulations relating to the teaching of chemistry for medical degrees of the University of London, the following resolutions have been approved by the undersigned, being the recognised teachers of chemistry in the twelve medical schools of the University. They believe that these recommendations can be carried into effect without lengthening the medical course.

1. That instruction in physiological and pathological chemistry can in no sense be regarded as taking the place of instruction in the pure scientific organic chemistry hitherto required by the University.
2. That this instruction in organic chemistry can only properly be given in a chemical laboratory and by a chemist.
3. That a careful scientific study of organic chemistry is essential for any later intelligent study of physiological and pathological chemistry.
4. That this study of organic chemistry must be preceded by a study of inorganic chemistry.
5. That to properly carry out such an education the Preliminary Scientific Course and Examination in Chemistry should be identical in scope with the revised Intermediate Course and Examination in Science.
6. That to provide for the proper study of organic chemistry a further period is required followed by a University examination conducted by chemists.
7. That the examination in organic chemistry should consist of a three hours' paper and three hours' practical work.
8. That it would be impossible for a student to take the examination in both inorganic and organic chemistry at the end of one academic year, unless he had previously studied inorganic chemistry.

As an indication of the amount of organic chemistry necessary, the teachers of chemistry in the medical schools of the University are prepared to submit a draft schedule adapted to medical requirements:—

Edward C. Cyril Baly (University), Hugh Candy (London), F. D. Chattaway (St. Bartholomew's), Arthur W. Crossley (St. Thomas's), Clare de B. Evans (Royal Free), J. Addyman Gardner (St. George's), H. Wilson Hake (Westminster), A. M. Kellas (Middlesex), H. Forster Morley (Charing Cross), John M. Thomson (King's), John Wade (Guy's), W. H. Willcox (St. Mary's).

## C.—MEMORANDUM ON THE POSITION OF CHEMISTRY IN THE MEDICAL CURRICULUM.

(Being a Reply to the Recent Report of the Board of Intermediate Medical Studies of the University of London).

The recent changes in the regulations relating to the medical degrees of the University of London, whereby the study of Organic Chemistry has been relegated to the preliminary scientific year, and must therefore be taken concurrently with Inorganic and General Chemistry, Experimental Physics, and General Biology, render it impossible for the student to acquire that knowledge of the scientific chemical basis of Physiology and Pathology, which has hitherto been ensured by the inclusion of Organic Chemistry in the Intermediate Examination in Medicine.

The reasons for thus lowering the standard of scientific medical education appear to be summarised in a report which has recently been formulated by the Board of Intermediate Medical Studies (October 27th, 1903), from which the following excerpt is taken :—

"This Board is strongly of opinion that the student of Medicine should complete his study of Pure Chemistry before beginning Anatomy and Physiology. Hitherto the study of these fundamental medical sciences has been hampered and injuriously limited by the amount of time, always considerable and of late years increasing, necessarily devoted to Organic Chemistry. Nevertheless, that subject has always offered great difficulties to the student, and it is within the knowledge of many teachers that rejections in Chemistry at the Intermediate Examination in Medicine have commonly been more numerous than in either Anatomy or Physiology. The subject of Chemistry has overweighted the Intermediate Examination, and has been a great obstacle to the attainment of the Degree of the University. This Board is therefore resolutely opposed to the reinstatement of Chemistry in the Intermediate Course.

"It is further to be observed that, under the existing arrangement, the study of Chemistry by the student of Medicine does not cease with the Preliminary Scientific Examination. The application of Chemistry to Biological phenomena and processes forms a large part of the subjects of Physiology and Pathology. The special branches of Physiological Chemistry in the Intermediate Course and of Pathological Chemistry in the Advanced Course have now been greatly extended, and much practical work in these subjects has been introduced. In this way the student will obtain his training in Medical Chemistry; and he is no longer dependent on the Course of Pure Chemistry alone for his education in this subject. The position occupied by Pure Chemistry in the present medical curriculum is entirely that of a Preliminary Scientific study, and in no sense a Professional Study. What is required from the pure Chemist for medical students is, not to make them Organic Chemists, but to give them such an elementary grounding in Chemistry that they may be able to enter upon the study of the special branches of Physiological and Pathological Chemistry.

"Under these circumstances this Board is of opinion that there should be no difficulty in including the required amount of Chemistry, together with the other subjects, in the one year's Preliminary Scientific work, and that the Preliminary Scientific Examination, in respect of Chemistry, should be put on the same level with the Intermediate Examination in Science."

It is noteworthy, however, that when this report came before the Faculty of Medicine (December 11th, 1903), it was "received" but not adopted, and it was resolved by this body, after careful consideration, "that while agreeing that the Student of Medicine should complete his study of Pure Chemistry before beginning Anatomy and Physiology, the Faculty is of opinion that one year is insufficient for its proper study."

In discussing the position of Chemistry in the Medical curriculum, no one at the present day who is conversant

with modern Physiology and Pathology can fail to realise that the association of Chemistry, and especially of Organic Chemistry, with these subjects is constantly becoming closer; the Board of Intermediate Studies does not indeed deny this. An advanced knowledge of Pure Organic Chemistry of a special type is essential for the intelligent pursuit of Physiology and Pathology, and it is to be presumed that it is seriously proposed by this Board that instruction in this necessary Organic Chemistry should form part of the Physiological Course. It is, however, impossible for a teacher who is engaged in Physiological research to remain familiar with the difficulties encountered by the student of Organic Chemistry; for although the Physiologist during his studentship has become acquainted with the fundamental facts of this Science, he cannot possibly retain that intimacy with them which comes as a matter of course to the Chemist.

The Advanced Organic Chemistry on which Physiology is based, and which has hitherto been studied by the Intermediate student in Medicine, differs widely from a course which would be suited to the requirements of students of Pure Chemistry. Numerous sections of great Chemical interest, covering a vast extent of ground, such as the coal-tar dyes, and the derivatives of naphthalene and anthracene, are entirely omitted, as having no immediate bearing on Physiology and Pathology, whilst other sections, such as the Chemistry of the nitrogen compounds and carbohydrates, are needed to an extent which would not be warranted by their purely Chemical interest. It is not correct, therefore, to say that the Organic Chemistry taught under the former regulations as part of the Intermediate Course in Medicine was in no sense a professional study; it was essentially professional in character, and would not have suited any but medical students.

Organic Chemistry is based on Inorganic Chemistry, and cannot be taught to students having no previous knowledge of this division of the subject; hence the teaching of the two branches cannot be included in one academic year, if it is to be of University standard. Even when a University standard is not demanded, it is recognised that no more than the rudiments of Organic Chemistry can be taught together with Inorganic Chemistry during this period. In their recent Report on the various "qualifying" medical examinations, the Visitors of the General Medical Council, after dwelling on "the great and growing importance in Medicine of an acquaintance with the carbon compounds," remark that "the subject is so large, that only the outer fringe of it can be touched in the first year of medical study."

Organic Chemistry most emphatically has not "over-weighted the Intermediate Examination," nor "been a great obstacle to the attainment of the degree of the University," as the Board of Intermediate Studies maintains; at some of the largest of the London medical schools, where a record has been kept, the rejections have not been greater than those in Anatomy and Physiology, but such as might reasonably be expected where students are allowed to present themselves without selection. Not only is the subject not detrimental, but it constitutes in the opinion of most authorities a valuable complement in training to the study of the other Intermediate subjects.

It has been stated that sufficient Organic Chemistry for Medical students is included in the Intermediate Examination in Science. The amount in this syllabus, however, is merely nominal, and quite inadequate as a foundation for Physiological Chemistry. Hence the Intermediate Examination in Science should not be accepted as at present, as exempting the Medical student from the further study of Organic Chemistry. The former Preliminary Scientific Examination was substantially identical with the present Intermediate Examination in Science, and contained quite as much Chemistry as could be assimilated in an academic year by a student having to devote two-thirds of his time to Physics and Biology; there appears to be no reason why the two examinations should not again become

identical, as the Board of Intermediate Studies proposes, so far as Inorganic Chemistry is concerned.

It has been suggested that the Chemistry of the Matriculation Examination might be accepted in place of the Inorganic Chemistry of the Preliminary Scientific Examination. A Matriculation Course, however, should aim rather at the inculcation of scientific method, than at teaching the essential facts of Chemistry, and can in no sense be regarded as equivalent to the systematic study and Laboratory work required for the Preliminary Scientific Examination.

As more than one year is necessary—and this is the opinion not only of the Chemists and many Physiologists, but also of the Faculty of Medicine—it follows that if the student is to complete his study of Chemistry before beginning the two years' course of Anatomy and Physiology, the curriculum will extend over more than five years from Matriculation. There is, however, no sufficient reason why the study of Organic Chemistry should not, as formerly, run concurrently with that of Anatomy and Physiology during the first year of Intermediate studies. The Preliminary Scientific year is hopelessly overweighted by the recent regulations, and such students as may pass the examination in one year under present conditions will have no real grasp of their work; it is impossible for any student, however capable, without a previous knowledge of Chemistry to work through the present syllabus under eighteen months.

If, however, the Preliminary Scientific Examination were made identical with the Intermediate Examination in Science, it could be passed easily after one year's study; and if a subsequent special course in Organic Chemistry were arranged, forming as far as possible an introduction to Physiological and Pathological Chemistry, and followed by a University Examination, the standard would be maintained without lengthening the curriculum or overburdening the students. This Examination could be held at the same times as the Intermediate Examination in Medicine, but candidates, instead of being compelled to wait two years as under the former regulations, might be allowed to present themselves for it at any time after passing in the Chemistry of the Preliminary Scientific Examination or Intermediate Examination in Science.

P. Phillips Bedson (Newcastle), Hugh Candy (London), F. D. Chattaway (St. Bartholomew's), Arthur W. Crossley (St. Thomas's), Clare de B. Evans (Royal Free), Percy F. Frankland (Birmingham), J. Addyman Gardner (St. George's), H. Wilson Hake (Westminster), A. M. Kellas (Middlesex), H. Forster Morley (Charing Cross), John M. Thomson (King's), John Wade (Guy's), W. H. Willcox (St. Mary's), W. Carleton Williams (Sheffield).

January 21th, 1904.

**D.—COPY OF THE ORIGINAL MEMORIAL ADDRESSED TO THE VICE-CHANCELLOR IN 1901, WHEN THE CHANGES WERE IN CONTEMPLATION.**

WE, the undersigned Teachers of Chemistry in the University of London, and others interested in Medical Education, having heard that proposals to exclude Organic Chemistry from its present position in the Medical curriculum, or to exclude it entirely in favour of Chemical Physiology, are to be brought before the Senate, beg leave to represent:—

1. That the resolution to exclude Organic Chemistry from the Intermediate M.B. Examination was arrived at by a Board of *eleven* members, of whom only *one* represented Chemistry, and he was opposed to the resolution arrived at.

2. That the exclusion of Organic Chemistry, as taught by recognised teachers of Chemistry, from the curriculum of the medical student would be a retrograde step, would involve a lowering of the value of the London Degree, and would be contrary to the practice of other Universities. Chemical Physiology cannot be taught without a previous knowledge of Organic Chemistry.

3. That it is impossible to include Organic Chemistry, which is a necessary introduction to Physiology and Pathology, in the course of chemical teaching given for the Preliminary Scientific Examination.

4. That it is impossible to insure that a competent knowledge of Inorganic Chemistry can be acquired at the stage at which a candidate matriculates.

5. That the subject of Organic Chemistry should be retained in the Intermediate M.B. Examination, and that the Examination in this subject be taken at the end of the year following that in which the Preliminary Scientific Examination is passed.

6. That this compromise secures that the subject of Organic Chemistry will occupy only part of one year of the Medical curriculum, instead of two, as at present, and that Elementary Anatomy or Physiology can be studied concurrently with Organic Chemistry.

This Memorial was signed by—

Prof. Clifford Allbutt, F.R.S.; Prof. H. E. Armstrong, F.R.S.; Dr. Buckmaster; Mr. Hugh Candy; Dr. A. W. Crossley; Prof. Wyndham Dunstan, F.R.S.; Prof. Percy Frankland, F.R.S.; Mr. Addyman Gardner; Prof. Francis Gotch, F.R.S.; Mr. C. E. Groves, F.R.S.; Dr. Wilson Hake; Dr. A. M. Kellas; Prof. Liveing, F.R.S.; Dr. Moore; Dr. Forster Morley; Prof. Odling, F.R.S.; Mr. F. J. M. Page; Dr. Phillips, F.R.S.E.; Prof. W. Ramsay, F.R.S.; Prof. Emerson Reynolds, F.R.S.; Dr. W. J. Russell, F.R.S.; Mr. Chas. Slater; Dr. Thos. Stevenson; Prof. W. A. Tilden, F.R.S.; Prof. J. M. Thomson, F.R.S.; Mr. John Wade; Mr. W. H. Willcox; Prof. Carleton Williams; Dr. George Young.

## PROCEEDINGS OF SOCIETIES.

### CHEMICAL SOCIETY.

*Ordinary Meeting, Wednesday, January 20th, 1904.*

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MESSRS. U. O. S. Nairne and J. T. Hall were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. J. P. Ackroyd, B.Sc., 116, Richmond Street, Accrington, Lancs.; Charles Thomas Bennett, 57, Larkhall Rise, Clapham, S.W.; George E. P. Broderick, B.Sc., Lampeter, South Wales; Julian Drugman, Ph.D., La Bocca, Cannes, France; George Fry, Carlin Brae, Berwick-on-Tweed; Francis D'Oyley Mears, jun., 4, Nyanza Terrace, Swansea; Jatindranath Sen, M.A., 71, Cathedral Mission Lane, Calcutta; Raymond Louis Siau, 15, Merridale Lane, Wolverhampton; Henry E. Stevenson, Avondale, Ditton Hill, Surrey; Charles H. Thompson, Hillcroft, Amblecote, Stourbridge; Hubert Thompson, B.Sc., Holmes Chapel, Cheshire; Léon Edward Walling, 43, Union Road, Rotherhithe; Charles Edward Whiteley, 9, Abyssinia Grove, Leeds; William Henry Woodcock, 10, Chesson Road, West Kensington, W.

Certificates in favour of the following were authorised by the Council for presentation for ballot under By-law I. (3):—Anu Ghose, 42, Shambazar Street, Calcutta; J. P. Montgomery, Agricultural College, Starkville, Miss., U.S.A.

THE PRESIDENT announced that since the last meeting of the Society the Rev. T. J. Prout, M.A., F.G.S., Senior Student of Christ Church, Oxford, had presented to the Society a photograph of a portrait by Hayes of his father, Dr. William Prout, F.R.S., which was considered to be a very good likeness of the author of the famous paper "On the Relation between the Specific Gravities of Bodies in

their Gaseous State and the Weights of their Atoms," published anonymously in Thomson's *Annals of Philosophy* in November, 1815.

The Council has ordered the following report to be printed in the Journal and in the Proceedings of the Society:—

#### REPORT OF THE INTERNATIONAL COMMITTEE ON ATOMIC WEIGHTS.

The International Committee on Atomic Weights\* has the honour to offer the following report:—

In the table of atomic weights for 1904, only two changes from 1903 are recommended. The atomic weight of caesium has been slightly modified to accord with the recent determinations by Richards and Archibald, and that of cerium in conformity with the measurements by Brauner. The value for lanthanum is still in controversy, and any change here would therefore be premature. The same consideration may also be urged with regard to iodine. Ladenburg has shown that the accepted number for iodine is probably too low, but other investigations upon the subject are known to be in progress, and until they have been completed it would be unwise to propose any alteration.

#### International Atomic Weights. (1904)

|                    |    | O=16.  | H=1    |
|--------------------|----|--------|--------|
| Aluminium .. .. .  | Al | 27.1   | 26.9   |
| Antimony .. .. .   | Sb | 120.2  | 119.3  |
| Argon .. .. .      | A  | 39.9   | 39.6   |
| Arsenic .. .. .    | As | 75.0   | 74.4   |
| Barium .. .. .     | Ba | 137.4  | 136.4  |
| Bismuth .. .. .    | Bi | 208.5  | 206.9  |
| Boron .. .. .      | B  | 11     | 10.9   |
| Bromine .. .. .    | Br | 79.96  | 79.36  |
| Cadmium .. .. .    | Cd | 112.4  | 111.6  |
| Cæsium .. .. .     | Cs | 132.9  | 131.9  |
| Calcium .. .. .    | Ca | 40.1   | 39.8   |
| Carbon .. .. .     | C  | 12.00  | 11.91  |
| Cerium .. .. .     | Ce | 140.25 | 139.2  |
| Chlorine .. .. .   | Cl | 35.45  | 35.18  |
| Chromium .. .. .   | Cr | 52.1   | 51.7   |
| Cobalt .. .. .     | Co | 59.0   | 58.56  |
| Columbium .. .. .  | Cb | 94     | 93.3   |
| Copper .. .. .     | Cu | 63.6   | 63.1   |
| Erbium .. .. .     | Er | 166    | 164.8  |
| Fluorine .. .. .   | F  | 19     | 18.9   |
| Gadolinium .. .. . | Gd | 156    | 155    |
| Gallium .. .. .    | Ga | 70     | 69.5   |
| Germanium .. .. .  | Ge | 72.5   | 71.9   |
| Glucium .. .. .    | Gl | 9.1    | 9.03   |
| Gold .. .. .       | Au | 197.2  | 195.7  |
| Helium .. .. .     | He | 4      | 4      |
| Hydrogen .. .. .   | H  | 1.008  | 1.000  |
| Indium .. .. .     | In | 114    | 113.1  |
| Iodine .. .. .     | I  | 126.85 | 125.90 |
| Iridium .. .. .    | Ir | 193.0  | 191.5  |
| Iron .. .. .       | Fe | 55.9   | 55.5   |
| Krypton .. .. .    | Kr | 81.8   | 81.2   |
| Lanthanum .. .. .  | La | 138.9  | 137.9  |
| Lead .. .. .       | Pb | 206.9  | 205.35 |
| Lithium .. .. .    | Li | 7.03   | 6.98   |
| Magnesium .. .. .  | Mg | 24.36  | 24.18  |
| Manganese .. .. .  | Mn | 55.0   | 54.6   |
| Mercury .. .. .    | Hg | 200.0  | 198.5  |
| Molybdenum .. .. . | Mo | 96.0   | 95.3   |
| Neodymium .. .. .  | Nd | 143.6  | 142.5  |
| Neon .. .. .       | Ne | 20     | 19.9   |
| Nickel .. .. .     | Ni | 58.7   | 58.3   |
| Nitrogen .. .. .   | N  | 14.04  | 13.93  |
| Osmium .. .. .     | Os | 191    | 189.6  |
| Oxygen .. .. .     | O  | 16.00  | 15.88  |
| Palladium .. .. .  | Pd | 106.5  | 105.7  |

\* The original members of the Committee take great pleasure in announcing the addition to their number of Professor Henri Moissan. They are confident that this increase will meet with universal approval.

|                      |    | O=16.  | H=1.   |
|----------------------|----|--------|--------|
| Phosphorus .. .. .   | P  | 31.0   | 30.77  |
| Platinum .. .. .     | Pt | 194.8  | 193.3  |
| Potassium .. .. .    | K  | 39.15  | 38.86  |
| Praseodymium .. .. . | Pr | 140.5  | 139.4  |
| Radium .. .. .       | Rd | 225    | 223.3  |
| Rhodium .. .. .      | Rh | 103.0  | 102.2  |
| Rubidium .. .. .     | Rb | 85.4   | 84.8   |
| Ruthenium .. .. .    | Ru | 101.7  | 101.0  |
| Samarium .. .. .     | Sm | 150    | 148.9  |
| Scandium .. .. .     | Sc | 44.1   | 43.8   |
| Selenium .. .. .     | Se | 79.2   | 78.6   |
| Silicon .. .. .      | Si | 28.4   | 28.2   |
| Silver .. .. .       | Ag | 107.93 | 107.12 |
| Sodium .. .. .       | Na | 23.05  | 22.88  |
| Strontium .. .. .    | Sr | 87.6   | 86.94  |
| Sulphur .. .. .      | S  | 32.06  | 31.83  |
| Tantalum .. .. .     | Ta | 183    | 181.6  |
| Tellurium .. .. .    | Te | 127.6  | 126.6  |
| Terbium .. .. .      | Tb | 160    | 158.8  |
| Thallium .. .. .     | Tl | 204.1  | 202.6  |
| Thorium .. .. .      | Th | 232.5  | 230.8  |
| Thulium .. .. .      | Tm | 171    | 169.7  |
| Tin .. .. .          | Sn | 119.0  | 118.1  |
| Titanium .. .. .     | Ti | 48.1   | 47.7   |
| Tungsten .. .. .     | W  | 184.0  | 182.6  |
| Uranium .. .. .      | U  | 238.5  | 236.7  |
| Vanadium .. .. .     | V  | 51.2   | 50.8   |
| Xenon .. .. .        | Xe | 128    | 127    |
| Ytterbium .. .. .    | Yb | 173.0  | 171.7  |
| Yttrium .. .. .      | Yt | 89.0   | 88.3   |
| Zinc .. .. .         | Zn | 65.4   | 64.9   |
| Zirconium .. .. .    | Zr | 90.6   | 89.9   |

Many of the atomic weights given in the table are well known to be more or less uncertain. This is especially true with respect to the rarer elements, such as gallium, indium, columbium, tantalum, &c. But some of the commoner elements also stand in need of revision, and we venture to call attention to a few of these. Among the metals, the atomic weights of mercury, tin, bismuth, and antimony should be re-determined, for the reason that the existing data are not sufficiently concordant. Palladium also, on account of discrepancies between different observers, and possibly vanadium, for which the data are too few, deserve some attention. Among the non-metals, phosphorus has been peculiarly neglected, and our knowledge of the atomic weight of silicon rests upon a single ratio. In the latter case, confirmatory data are much to be desired. Upon any of these elements, new investigations would be most serviceable.

There is one other point to which we may properly call attention. Many of the ratios from which atomic weights have been calculated were measured in vessels of glass, by processes involving the use of strong acids. In such cases, the solubility of the glass becomes an important consideration, even when no transfer of material from one vessel to another has occurred. A slight conversion of silicate into chloride would cause an increase of weight during the operation, and so introduce an error into the determination. Such errors are doubtless very small, but still they ought not to be neglected. Now that vessels of pure silica, the so-called quartz-glass, are available for use, they might well replace ordinary glass in all processes for the determination of atomic weights. An investigation into the relative availability of the two kinds of glass is most desirable.

F. W. CLARKE  
T. E. THORPE  
K. SEUBERT  
HENRI MOISSAN } Committee.

Of the following papers, those marked \* were read:—

\*1. "The Chemical Reactions of Nickel Carbonyl. Part I. Reactions with the Halogens and other Inorganic Substances." By JAMES DEWAR and HUMPHREY OWEN JONES.

The previous study of the physical properties of nickel carbonyl (*Proc. Roy. Soc.*, 1903, lxi., 627) showed that it was a comparatively stable substance when heated under pressure. The authors have undertaken the investigation of its chemical behaviour mainly from a thermochemical standpoint, with the view of studying its constitution and its possible use as a synthetical agent.

Nickel carbonyl is completely decomposed by solutions of chlorine, bromine, iodine, cyanogen, and sulphur in organic solvents, carbon monoxide and a nickel compound being produced; in no case was any combination of the carbon monoxide with the halogen or other reagent observed, even when a considerable excess of the latter was present.

From Mittasch's value (52.2 Cal.) for the heat of formation of nickel carbonyl and Thomsen's values for nickel compounds, it was to be expected that chlorine and bromine would decompose it readily, but that iodine, cyanogen, and sulphur would not. In the case of iodine, the heat necessary to carry out the reaction appears to be obtained by the formation of molecular complexes of nickel iodide and the solvent (for example, ether and chloroform), and in the case of sulphur by the formation of a higher sulphide, probably  $\text{Ni}_2\text{S}_3$ . Liquid chlorine and bromine decompose solid nickel carbonyl, but solid iodine appears to have no action on liquid nickel carbonyl.

Iodine mono- and tri-chlorides and cyanogen iodide in carbon tetrachloride solution react with nickel carbonyl in two distinct stages, the free iodine liberated at first subsequently decomposing a further quantity of nickel carbonyl.

Hydrogen iodide readily interacts with nickel carbonyl, whereas the corresponding bromide and chloride do not affect it.

Hydrogen sulphide reacts very slowly with nickel carbonyl, producing nickel monosulphide, hydrogen and carbon monoxide. Sulphuric acid also decomposes the compound slowly, giving rise to nickel sulphate, hydrogen, and carbon monoxide.

\*2. "The Chemical Reactions of Nickel Carbonyl. Part II. Reaction with Aromatic Hydrocarbons in Presence of Aluminium Chloride. Synthesis of Aldehydes and Anthracene Derivatives." By JAMES DEWAR and HUMPHREY OWEN JONES.

Nickel carbonyl does not react with either aluminium chloride or benzene separately, but with a mixture of the two substances a violent reaction begins immediately and torrents of hydrochloric acid are evolved. The reaction with various benzene homologues has been investigated both at the ordinary temperature and also at  $100^\circ$ .

From benzene at the ordinary temperature, benzaldehyde is produced together with traces of oils having high boiling points. At  $100^\circ$ , the quantity of benzaldehyde is much smaller, and anthracene, which is now the chief product, is produced in considerable quantities. The mechanism of this production of anthracene from benzaldehyde has not been elucidated, but the reduction is probably due to the action of the nickel produced by the decomposition of the nickel carbonyl.

Toluene gives *p*-tolualdehyde and 2:6-dimethylanthracene (m. p.  $215-216^\circ$ ). *m*-Xylene similarly yields 2:4-dimethylbenzaldehyde and a tetramethylanthracene melting at  $280^\circ$ , which is in all probability 1:3:5:7 tetramethylanthracene. Mesitylene yields an aldehyde only, condensation to an anthracene derivative being in this case impossible.

Naphthalene behaves in an entirely different way; no aldehyde is formed either in the cold or at  $100^\circ$ , and a hydrocarbon,  $\text{C}_{16}\text{H}_{12}$ , is produced together with oily or resinous substances having very high boiling points, which appear to be further condensation products, since the quantity produced is much greater at the higher temperature. This hydrocarbon, which melts at  $180-181^\circ$ , is probably identical with that obtained by Bischoff by the

interaction of naphthalene and methyl chloride in the presence of aluminium chloride, and may also be identical with that produced from ruficoccine or coccine by distillation with zinc dust.

\*3. "Optically Active Asymmetric Nitrogen Compounds. *d*- and *l*-Phenylbenzylmethylethylammonium Salts." By HUMPHREY OWEN JONES.

The resolution of the phenylbenzylmethylethylammonium salts was undertaken with the view of showing that optical activity in nitrogen compounds was independent of the existence of isomerides of the compound in question.

The only optically active nitrogen compounds known at present are the *d*- and *l*- $\alpha$ -phenylbenzylmethylallylammonium salts, and these are also the only quinquivalent nitrogen compounds (not containing an asymmetric carbon atom) which have been shown to exist in definite stable isomeric forms.

Phenylbenzylmethylethylammonium iodide has been prepared in the three possible ways, namely, by the following combinations:—(1) methylethylaniline and benzyl iodide, (2) benzylethylaniline and methyl iodide, (3) benzylmethylaniline and ethyl iodide. The velocity of formation of the iodide is remarkably different in the three cases, that in the first combination being the greatest and that in the third being the least. The compound produced, which is the same in all three cases, crystallises from alcohol in lustrous, prismatic prisms, the melting point of which is about  $140-142^\circ$ , but this is to a certain extent dependent on the rate of heating.

The iodide was converted into the *d*- and *l*-camphorsulphonates by boiling molecular proportions of the salt and silver *d*- and *l*-camphorsulphonates with ethyl acetate and a small quantity of alcohol.

The camphorsulphonate was crystallised repeatedly from a mixture of dry ethyl acetate and ethylal at a comparatively low temperature until its rotatory power was constant. *d*-Phenylbenzylmethylethylammonium *d*-camphorsulphonate crystallises in lustrous prisms melting at  $180-181^\circ$ . It is very sparingly soluble in ethyl acetate and acetone and has  $[\text{M}]_D = 71.0^\circ$  in aqueous solution.

The corresponding *ll*-salt is identical with its *dd*-isomeride in appearance and properties and has  $[\text{M}]_D = -71.2^\circ$ . *d*-Phenylbenzylmethylethylammonium iodide, which was obtained by mixing the calculated quantities of potassium iodide and the *d*-camphorsulphonate in aqueous solution, has the same appearance, crystalline form, and melting point as the inactive iodide; it is very sparingly soluble in alcohol and other solvents. Its rotatory power is small, so that determinations of this constant are difficult, the observed  $[\text{M}]_D$  being about  $30^\circ$ . The corresponding *l*-iodide, which was prepared from the *l*-camphorsulphonate in a similar manner, had  $[\text{M}]_D = 30^\circ$  (approximately). The iodides undergo racemisation in chloroform solution, but their molecular weight in this solvent appears to be nearly normal at the ordinary temperature.

The inactive and laevorotatory bromides were also prepared and were found to melt at  $155-156^\circ$ . The latter compound has a higher specific rotatory power than the *l*-iodide.

The feeble rotatory power of these salts compared with that of the active  $\alpha$ -phenylbenzylmethylallylammonium is remarkable, since the only difference is the replacement of an allyl by an ethyl radicle.

\*4. "A Microscopic Method of Determining Molecular Weights." By GEORGE BARGER.

The author has continued the experiments of which a preliminary account has already been given (*Proc.*, 1903, xix., 121). The method is based on the comparison of the vapour pressures of two solutions, of which biconcave, lenticular drops are placed in a capillary tube. A difference in the vapour pressures causes a mutual variation in the size of the drops, which is observed by means of an eyepiece micrometer.



About one hundred determinations with various substances in eleven different solvents have now been made, and the experimental error is found to vary from 5 to 10 per cent. Solvents of widely differing boiling points have been employed, including ether, xylene, and light petroleum (b. p. 50–60°). Some determinations were performed with minute quantities of the dissolved substance.

The method is being applied to the study of association in mixtures of associative and non-associative solvents. The behaviour of benzoic acid in mixtures of benzene and ethyl alcohol, and of cinnamic acid in mixtures of chloroform and methyl alcohol, has so far been studied, the results showing that a small proportion of the alcohol suffices to give these acids a normal molecular weight. Should this phenomenon prove to be general, it would be of practical value in determining molecular weights in those cases where associative solvents only can be employed.

#### DISCUSSION.

Dr. PHILIP asked whether it would be possible to carry out the experiments at a higher temperature, nearer the boiling point of the solvent, in order to shorten the time necessary for the establishment of equilibrium between the drops.

Dr. HEWITT suggested the possibility of using two graduated tubes fitted with tightly-fitting india-rubber stoppers and connected by a T-tube, the third branch of which should have a stop-cock. Into one of the graduated tubes a standard solution could be introduced, and into the other a weighed quantity of the substance, together with some of the solvent. It would then only be necessary to exhaust the apparatus, place it in a bath until equilibrium was attained, allow to cool, and measure the respective volumes of solution in the two graduated tubes.

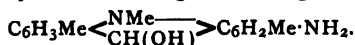
Mr. BARGER, in reply to Dr. Philip, said that the tubes must either be measured at the higher temperature or cooled down to that of the microscope. In the latter case, the solvent condenses between the drops on the walls of the tube, and thus produces an error. In the former case, it has not been found possible to prevent the condensation of water vapour on the frontal lens of the microscope objective.

Macroscopic methods similar to that suggested by Dr. Hewitt have been tried, both volumetrically and gravimetrically, but without the least success. The essential conditions which make the present method possible are.—

- (1) The accuracy with which extremely small changes in volume of the drops can be detected under the microscope.
- (2) The large evaporating surface presented by the drops, as compared with their volume.

\*5. "Studies in the Acridine Series." Part I. By JOHN JACOB FOX and JOHN THEODORE HEWITT.

The authors find that 6-acetamino-2:7-dimethylacridine (m. p. 270° uncorr., Ullmann gives 258°, *Ber.*, 1903, xxxvi., 1025) unites with methyl iodide to form a quaternary acridinium iodide, a substance yielding a precipitate with ammonia. The isolation of the corresponding carbinol seems impracticable on account of the ease with which the acetyl group is hydrolysed. To ensure complete hydrolysis, the solution obtained on boiling the precipitate with dilute sulphuric acid is treated with ammonia, when the base is liberated. This compound, which, when crystallised from acetone, has a pale red colour, melts at 210° (uncorr.) and corresponds with the formula  $C_{16}H_{18}ON_2$ ; it is probably a carbinol having the following constitution:—

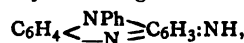


When the carbinol base is heated for some hours at 184°, partial dehydration occurs, and on warming the nitrobenzene solution of the substance, a very appreciable evolution of water is observed. On filtering the hot

solution and subsequently treating the cooled filtrate with light petroleum, a dark red compound,  $C_{16}H_{14}N_2$ , is deposited, which is readily soluble in acids and probably has the constitution—



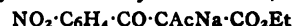
because the oxygen removed as water was attached in the carbinol base to the carbon atom of the median ring. Such a formula for the anhydro-base agrees with the constitution—



for the base of aposafranine, and does not correspond with the aposafranine formula suggested by Kehrmann (com. *pare Ber.*, 1896, xxix., 2316; *Annalen*, 1896, ccxc., 256).

6. "o-Nitrobenzoylactic Acid." By EDWARD RUSHTON NEEDHAM and WILLIAM HENRY PERKIN, jun.

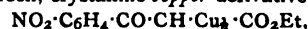
Ethyl sodioacetacetate (2 mols.) reacts readily with o-nitrobenzoyl chloride (1 mol.), yielding the sodium compound of ethyl o-nitrobenzoylacetate,



(Gevekoth, *Annalen*, 1883, ccxxi., 323), and when this product is digested with ammonia and ammonium chloride (compare Claisen, *Annalen*, 1896, ccxc., 67) the acetyl group is eliminated and ethyl-o-nitrobenzoylacetate,



is produced. This ester is a pale brown oil which, in alcoholic solution, gives an orange-red colouration with ferric chloride; it readily dissolves in dilute aqueous caustic potash, yielding a yellow, crystalline, potassium derivative,  $NO_2 \cdot C_6H_4 \cdot CO \cdot CHK \cdot CO_2Et$ , and when its ethereal solution is shaken with ammoniacal copper sulphate, the green, crystalline copper derivative,



is obtained. Ethyl o-nitrobenzoylacetate dissolves in concentrated sulphuric acid, and if the solution is heated at 90° for a few minutes and then poured into water, o-nitrobenzoylactic acid,  $NO_2 \cdot C_6H_4 \cdot CO \cdot CH_2 \cdot CO_2H$ , separates, and may be purified by crystallisation from water. It melts at about 118°, and when boiled with water is decomposed into o-nitroacetophenone and carbon dioxide.

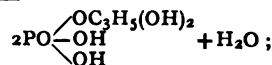
(To be continued).

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

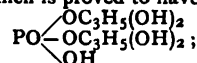
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 1, January 4, 1904.

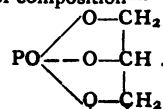
Phosphoric Ethers of Glycerin.—P. Carré.—In a preceding paper the author shows that the etherification of phosphoric acid by glycerin gives rise to three ethers. He now investigates—(1) The glycerophosphoric acid, which on heating is partially decomposed into phosphoric acid and a diether,—



(2) the diether, which is proved to have the composition—



and (3) the triether, of composition—



**Retrogradation and Coagulation of Amidon.**—L. Maquenne, A. Fernbach, and J. Wolff.—The authors find that starch coagulated by amyl-coagulase is like retrograde amidon, a complex mixture which is only partially saccharifiable—and that the diastatic coagulation of starch, judged from the nature of the resulting products, can be compared to the phenomenon described by one of the authors under the name of retrogradation. It can be distinguished by the rapidity with which the change takes place, the effect being to arrive almost instantaneously at the same result as would be observed when starch is left for a long time to itself.

**Sodium Monosulphide as an Indicator in the Estimation of Glucose by Fehling Solution.**—L. Beulaygue.—In order to assist in the measurement of the point of complete decolouration of Fehling's solution, and, consequently, the end of the reaction, the author finds certain indicating reagents of great value. For this purpose, monosulphide of sodium is used, dissolved in ten times its weight of distilled water. A drop of this substance on a piece of filter-paper acts as an indicator with the reduced Fehling solution, for when the liquid is not fully reduced a black or brown stain of copper sulphide is produced. This disappears directly reduction is complete, the filter-paper stain remaining colourless. The end of the reaction can in this manner be very sharply defined.

## MISCELLANEOUS.

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 1st inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. Mr. W. B. Byran, Mr. C. E. Challis, Mr. A. F. H. Dick, Mr. W. Gowland, Sir Charles Hartley, K.C.M.G., Miss C. Jones, Mr. C. E. Layton, Mr. C. F. Lan-Davis, Mr. H. Loeffler, Brigade Surgeon Lieut.-Col. C. W. MacRury, Mr. W. M. Ogilvie, Mr. C. G. P. Pownall, Lady Priestley, Mr. C. F. Rousselet, R. E. Tatham, and Mr. J. Weir were elected members. The special thanks of the Members were returned to Sir Andrew Noble, Bart., Dr. Frank McClean, Mr. J. B. Brown-Morison, J.P., and Lord Greenock for their donations to the Fund for the Promotion of Experimental Research at Low Temperatures.

**Chemical, Metallurgical, and Mining Society of South Africa.**—Annual Competition.—It has been decided to make six awards annually of £50 each, accompanied by a gold medal and diploma, for the following subjects:—(1) Mining; (2) Milling; (3) Cyaniding; (4) Chemistry, Pure and Applied; (5) Metallurgy (other than Milling and Cyaniding); (6) Agricultural Chemistry. The following are the conditions:—

- In awarding the prizes submitted for competition by members, associates, and students, all papers other than those by hon. members to be considered.
- The above awards to be made by specially constituted Committees, consisting of three members of the Society for each class, to be elected at a Special General Meeting of the Society, such Committees having power to refer selected papers to one or more competent authorities. The awards to go by a majority of votes and to be confirmed by the Council.
- No one to be awarded the prize in the same class two consecutive years.
- During the first year, ending June, 1904, no member of the Council may receive the cash prize, but may be awarded a Society's medal and diploma. In the event of any one of the Council being credited with the best contribution in any class, such member of Council would be awarded a Society's medal and diploma, whilst the cash prize, accompanied by a diploma, would fall to the ordinary member giving the next best paper in the same class.

- No one to be eligible for more than two prizes or two medals in the same year.
- In awarding the prizes, papers dealing with or having application to the South African Mining Industry to have preference. In the event of the prize awarding Committees considering that any paper submitted is not of sufficient interest to merit a prize, the prize may be withheld.
- No competitor to be allowed to adjudicate upon his own paper.
- In the event of circumstances arising such as are not provided for in the above conditions the Council shall have power to deal with the same.

A sixth prize of £50, accompanied by the Society's special gold medal and diploma, for the paper of the greatest utility to the Mining Industry, shall be open to the whole membership of the Society, and the adjudicating Committee shall consist of those members of the Council who do not compete, with power to take advice from expert authorities.

**Yellow Arsenic.**—MM. Erdmann and V. Unruh.—The authors have undertaken the examination of the yellow modification of arsenic already mentioned by other chemists. This yellow modification of arsenic, which corresponds to white phosphorus, is prepared by cooling arsenical fumes very rapidly. The sublimation is effected by heating arsenic in a tube of aluminium at the ordinary pressure. The fumes are carried in a current of an inert gas, such as carbonic acid. The arsenic must be heated to above 360°, and the fumes condensed very rapidly by very energetic cooling. By condensing the fumes at 360° we obtain metallic arsenic; by condensing at about 210° we obtain a black powder. As the yellow arsenic is further very rapidly transformed in the solid state into the black modification, we collect it in bisulphide of carbon, which dissolves the yellow variety. Finally, the whole of the apparatus must be protected from the action of light, which facilitates the transformation of the yellow arsenic into ordinary arsenic. See the original article for the apparatus used by the authors. The sulpho-carbonic solution, concentrated to saturation, and cooled in the dark in a mixture of CO<sub>2</sub> and ether at a temperature of at least -70°, leaves a deposit of arsenic in the form of a yellow powder. This yellow arsenic will keep without change in the dark at a temperature of -65° to -70°. Its solubility in bisulphide of carbon for 100 c.c. of the solvent is:—

| Temperature. | Arsenic.  |
|--------------|-----------|
| +46°         | 11'0      |
| +18—20°      | 7'5—8'0   |
| +12°         | 5'5—6'0   |
| +0°          | 3'8—4'0   |
| -15°         | 2'0—2'5   |
| -60°         | 0'8—1'0   |
| -80—85°      | insoluble |

It is slightly soluble in benzene, and still less so in acetic ether. In these sulpho-carbonic solutions the authors have also observed the formation of a precipitate of a brownish red colour adhering as a rule to the sides of the vessel; this represents a third modification of arsenic. This modification is stable when exposed to light, and recalls the red modification of phosphorus. The brownish-red arsenic has not been obtained entirely free from sulphur; it is very probably identical with the brown arsenic observed by Geuther (*Zeitsch. Zeit. f. Medizin. u. Naturw.* vol. x., suppl. p. 123), and obtained by him by the reduction of arsenious acid with trichloride of phosphorus. The molecular weight of the yellow arsenic, determined by Erdmann and Unruh's method (*Zeit. Anorg. Ch.*, vol. xxxii., p. 413), corresponds to As<sub>4</sub>.—*Zeit. Anorg. Chem.*, vol. xxxii., p. 437.

**Some Salts of Cadmium.**—H. Grossmann.—This paper deals with the thiocyanates which have been also examined by MM. Meitzendorf (*Pogg. Ann.*, vol. lvi., p. 81) and Walden (*Zeit. Anal. Chem.*, vol. xxxiii., p. 373).

To obtain the simple salt  $(\text{CyS})_2\text{Cd}$ , it is best to react with equal molecules of  $\text{SO}_4\text{Cd}$  and  $(\text{CyS})_2\text{Ba}$  (obtained from  $\text{CySNH}_4$  and  $\text{Ba}(\text{OH})_2$ ; filter, and on evaporation we obtain crystalline crusts. If we boil a solution of  $\text{CySNH}_4$  with  $\text{Cd}(\text{OH})_2$  recently precipitated and freshly washed,  $\text{NH}_3$  is given off, and the oxide is dissolved; after prolonged boiling, filtration, and evaporation on the water-bath, we obtain a liquid which, in the desiccator, gives fairly large but easily decomposed crystals of the mono-ammoniacal salt,  $(\text{CyS})_2\text{Cd} \cdot \text{NH}_3$ , in clinorhombic prisms,  $m \text{ } h^1 g^1$ ;  $m \text{ } m = 104^\circ$ ;  $m \text{ } p = 107^\circ 45'$ ; cleavages,  $h^1$ ,  $g^1$ . An excess of ammonia converts this salt into a diammoniacal salt,  $(\text{CyS})_2 \cdot 2\text{NH}_3$ , described by Meitzendorf. In these preparations there is also formed a double salt which is found in the mother-liquors,  $(\text{CyS})_2\text{Cd} \cdot 2\text{CySNH}_4 + 2\text{H}_2\text{O}$ ; it is deliquescent, fuses at  $25^\circ$ , and is insoluble in alcohol. It forms clinorhombic prisms,  $p$  dominant,  $m \text{ } h^1 a^1$ ,  $m \text{ } p = 77^\circ 22'$ ,  $p \text{ } h^1 = 98^\circ 56'$ . Other double salts, soluble and well crystallised, have also been prepared. Cadmico-potassic thiocyanate,  $(\text{CyS})_2\text{Cd} \cdot 2\text{CySK} + 2\text{H}_2\text{O}$ , in regular octahedra. Thiocyanate of rubidium,  $\text{CySRb}$ , in long needles similar to  $\text{CySK}$ , from  $\text{CO}_3\text{Rb}_2$  and  $\text{CySNH}_4$ . Thiocyanate of cadmium and rubidium,  $(\text{CyS})_2\text{Cd} \cdot 2\text{CySRb} + 2\text{H}_2\text{O}$ , in hexagonal plates. The double salt of sodium is  $(\text{CyS})_2\text{Cd} \cdot \text{CySNa} + 3\text{H}_2\text{O}$ , in regular hexagonal plates,  $p \text{ } m \text{ } h^1$ . For the double salt of barium, the slightly soluble simple salt of cadmium is first formed; the concentrated mother-liquors eventually give large deliquescent plates of  $(\text{CyS})_2\text{Cd} \cdot 4(\text{CyS})_2\text{Ba} + 10\text{H}_2\text{O}$ . —*Berichte*, vol. xxxvi., p. 2665.

## NOTES AND QUERIES.

\*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Ash in the Human Body.—Will any reader kindly quote the proportion of ash in the human body, giving authority?—X.

## MEETINGS FOR THE WEEK.

- MONDAY, 8th.—Society of Arts, 8. (Cantor Lectures). "Oils and Fate—their Uses and Applications," by J. Lewkowitsch, Ph.D., &c.
- TUESDAY, 9th.—Royal Institution, 5. "Development of Animals," Society of Arts, 4.30. "The Biology of Federation," by the Hon. Sir John Alexander Cockburn, K.C.M.G.
- WEDNESDAY, 10th.—Society of Arts, 8. "Thermit—its Application to Metallurgical Engineering," by Charles Vernon Boys, F.R.S.
- THURSDAY, 11th.—Royal Institution, 5. "Recent Research in Agriculture," by A. D. Hall, M.A. Society of Arts, 4.30. "Our Commercial Relations with Afghanistan," by Colonel Sir Thomas H. Holdich, K.E., K.C.M.G., K.C.I.E., C.B.
- FRIDAY, 12th.—Royal Institution, 9. "Westminster Abbey in the Early Part of the Seventeenth Century," by The Very Rev. J. A. Robinson, D.D. Physical, 8. (At the Royal College of Science, South Kensington). Annual General Meeting. Address by Dr. R. T. Glazebrook, F.R.S., President.
- SATURDAY, 13th.—Royal Institution, 3. "Culture and Sculpture," by Charles Waldstein, Litt.D.

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### CONTENTS.

| ARTICLES:—                                                                                                                                                                                                                                      | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| On the Colorimetric Determination of Small Quantities of Phosphoric Acid and of Silica, by F. T. Veitch .....                                                                                                                                   | 73   |
| On the Part played by Benzene in Poisoning by Coal-gas, by R. Stadelin, M.D. ....                                                                                                                                                               | 74   |
| Precipitation and Separation by Weak Organic Bases, by E. T. Allen .....                                                                                                                                                                        | 76   |
| Institute of Chemistry .....                                                                                                                                                                                                                    | 76   |
| PROCEEDINGS OF SOCIETIES—                                                                                                                                                                                                                       |      |
| CHEMICAL SOCIETY.—The <i>cis</i> - and <i>trans</i> -Modifications of <i>α</i> -Trimethylglutaconic Acid—Influence of Nuclear Substitution on the Rate of Oxidation of the Side-chain; I., Oxidation of the Mono- and Dichlorotoluenes—&c. .... | 80   |
| CORRESPONDENCE .....                                                                                                                                                                                                                            | 82   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                                                                                                                     | 82   |
| MISCELLANEOUS .....                                                                                                                                                                                                                             | 83   |
| MEETINGS FOR THE WEEK .....                                                                                                                                                                                                                     | 84   |

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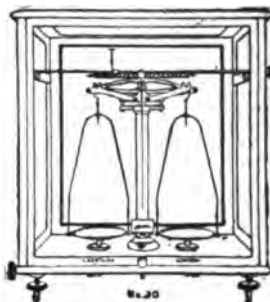
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2307.

## ON THE COLORIMETRIC DETERMINATION OF SMALL QUANTITIES OF PHOSPHORIC ACID AND OF SILICA.\*

By F. P. VEITCH.

In some of the field work of this bureau the analysis of drainage waters and of water extracts of soil is required daily. For this purpose the usual gravimetric processes are deemed impracticable on account of the time required, and it has been necessary to use more rapid volumetric, colorimetric, and photometric methods.

The writer suggested, and in the spring and early summer of the past year made an examination of, some known rapid colorimetric methods for the determination of phosphoric acid and of silica in these soil solutions. Incidentally, as the presence of iron in considerable amounts introduces errors in the determination of phosphoric acid, the rapid methods for the detection and estimation of minute quantities of iron have received some attention. As these latter methods have been thoroughly worked out and are quite well known, but passing reference need be made to them here.

For the estimation of phosphoric acid, the method described by Lepiere (*Bull. Soc. Chim.*, 1896, xv., 1213) and later studied in more detail by Woodman and Cayvan (*Journ. Am. Chem. Soc.*, xxiii., 96) promised to be the most rapid and to present the fewest difficulties in its manipulation.

For the adaptation of this method to the analysis of soil solutions, the influence of other compounds, both organic and inorganic, must be eliminated or determined. It was found that turbidity, organic matter where it produced a coloured solution, ammonia salts in large amounts, iron salts in small amounts, dissolved silica, and filter-paper introduce errors which are not negligible.

### Removal of Suspended Matter.

Turbidity may be removed by filtering through a Chamberland filter, which of course removes some of the phosphoric acid from the first runnings (see Table VIII.). It may also be quite largely, sometimes completely, removed by evaporation to dryness and filtering; or it may be corrected for by matching the standard phosphomolybdate solution against the turbid solution and subtracting this reading from the final readings. None of these procedures is perfectly satisfactory, but nothing better has yet been devised. All have been used here, according to the difficulties presented. Where filtration will remove all suspended matter, this procedure is to be preferred, but as this cannot always be accomplished, the Chamberland filter or evaporation must be resorted to.

In the work about to be described the reagents were prepared as directed by Woodman and Cayvan (*loc. cit.*) and were free from interfering substances. The same lot was used throughout the work, and all work was done at from 18°–22° C., the standard and the unknown solution being of course at sensibly the same temperature.

### Removing Organic Matter.

Organic matter which colours the solution at all must be removed, or it must be corrected for as above described, before reliable readings can be made. To accomplish this, two methods were tried: treatment with aqua regia, and ignition with magnesium nitrate. Several treatments with

aqua regia were required to insure the destruction of all organic matter, after which the solutions were carried to dryness, taken up with water and 2 c.c. nitric acid and compared in a reflecting colorimeter. The following readings were obtained:—

|   | Amount P <sub>2</sub> O <sub>5</sub> added. | Parts per Million. | Amount P <sub>2</sub> O <sub>5</sub> recovered. |
|---|---------------------------------------------|--------------------|-------------------------------------------------|
| 1 | 1.0                                         | 1.0                | 1.0                                             |
| 2 | 2.0                                         | 2.0                | 1.1                                             |
| 3 | 3.5                                         | 3.5                | 2.4                                             |
| 4 | 5.0                                         | 5.0                | 4.3                                             |
| 5 | 7.0                                         | 7.0                | 6.1                                             |
| 6 | 9.0                                         | 9.0                | 7.1                                             |

We see that there is considerable loss of phosphoric acid from this concentrated aqua regia solution, a fact previously pointed out by Woodman and Cayvan.

Other phosphate solutions were then treated with magnesium nitrate free from silica, evaporated to dryness with nitric acid, and ignited in a porcelain dish until all carbon was burned off. The residue was then taken up with water and nitric acid, and compared with the standard solution.

|    | Amount P <sub>2</sub> O <sub>5</sub> added. | Parts per Million. | Amount P <sub>2</sub> O <sub>5</sub> recovered. |
|----|---------------------------------------------|--------------------|-------------------------------------------------|
| 1  | 1.0                                         | 1.0                | 1.0                                             |
| 2  | 2.0                                         | 2.0                | 1.8                                             |
| 3  | 4.0                                         | 4.0                | 3.7                                             |
| 4  | 6.0                                         | 6.0                | 5.9                                             |
| 5  | 8.0                                         | 8.0                | 7.9                                             |
| 6  | 9.0                                         | 9.0                | 9.2                                             |
| 7  | 10.0                                        | 10.0               | 9.9                                             |
| 8  | 3.0                                         | 3.0                | 2.7                                             |
| 9  | 1.0                                         | 1.0                | 1.0                                             |
| 10 | 5.0                                         | 5.0                | 5.2                                             |
| 11 | 10.0                                        | 10.0               | 10.0                                            |

Average loss .. 0.06

The loss here is quite small, being as a rule within the limit of error of the reading. From these experiments it appears that the organic matter may be satisfactorily removed without loss of sensible amounts of phosphoric acid by ignition with magnesium nitrate and subsequent evaporation with nitric acid.\* It may be said, however, that when the colour due to organic matter is not greater than 0.2 to 0.3 part per million, it is hardly worth while to remove the organic matter, as a correction may be made for it by reading the colour due to organic matter against the standard, and correcting the final reading by the reading thus obtained.

The volatility of phosphoric acid upon evaporation of these water solutions is at first sight rather startling to analysts, and while there is not the slightest doubt that it does take place from solutions of phosphoric acid† and from disodium phosphate solutions, there appears to be no reason to think that it occurs from solutions containing sufficient bases to form the normal phosphates. In no case has the writer obtained a loss from solutions of this character. As phosphates dissociate readily, it seems possible that water extracts of soils may contain occasionally some free phosphoric acid or acid phosphates from which phosphoric acid is partly volatilised upon evaporation. That this does not take place in the presence of a large excess of base, even from acid solutions, is shown positively by the experiments with magnesium nitrate.

### Influence of the Presence of Other Salts.

Ammonia salts in considerable quantities decrease slightly the intensity of the colour reaction. Ammonium

\* Subsequent work has confirmed these results in every instance.

† It seems possible that this apparent loss may be due to the formation of pyrophosphoric acid.—*Ed. Am. Chem. Soc.*

\* From the *Journal of the American Chemical Society*, xxv., No. 2.

nitrate added to the unknown solution at the rate of 0.04 grm. per 50 c.c. gave readings on 4 parts per million of standard of only 3 parts, which increased a little on standing, but the solution soon precipitated. Ammonium chloride does not give a water-white solution always—has a tendency to be slightly opaque, and it also prevents the full development of the colour. The figures obtained are given in the table.

TABLE III.—Effect of Ammonium Salts.

| Parts per million $\text{NH}_4\text{NO}_3$ . | Parts per million $\text{P}_2\text{O}_5$ added to $\text{NH}_4\text{NO}_3$ solution. | = | Parts per million $\text{P}_2\text{O}_5$ without $\text{NH}_4\text{NO}_3$ . | Solution with $\text{NH}_4\text{NO}_3$ commenced to precipitate. |
|----------------------------------------------|--------------------------------------------------------------------------------------|---|-----------------------------------------------------------------------------|------------------------------------------------------------------|
| 4000                                         | 1.0                                                                                  | = | 0.7                                                                         | Not in five minutes.                                             |
| 4000                                         | 4.0                                                                                  | = | 2.0                                                                         | Nearly as dark, but precipitated too rapidly to be sure.         |
| 4000                                         | 4.0                                                                                  | = | 3.0                                                                         |                                                                  |
| 800                                          | 4.0                                                                                  | = | 3.3                                                                         | Precipitated in fifty minutes.                                   |
| 800                                          | 4.0                                                                                  | = | 3.7                                                                         | Precipitated in seventy-minutes.                                 |

Calcium nitrate, barium chloride, potassium nitrate, and aluminum sulphate, separately and together, were added to solutions at the rate of 0.25 grm. of each salt to 50 c.c. of solution. None of these except the aluminum sulphate affected the readings more than 0.1 part per million, or within the limit of error of readings. Aluminum sulphate in this quantity gave a dark, opaque solution which could not be matched satisfactorily against the standard.

It was found that iron salts in considerable quantities exerted a marked influence not only on the colour of the

solution itself, in the first instance, an influence which it was found could be corrected for by reading against the standard, but there is also a reaction between the iron salt and the molybdate, a reaction which appears to bear a relation to the amount of iron present, and which possibly may be used for the estimation of the iron in solution. As iron is present in many soil extracts, the reaction of iron with the molybdate solution required careful study in order to determine the amounts and conditions which would vitiate the phosphate readings. The results of the investigation of this point are given in Table IV. Unless otherwise stated, the two solutions were identical in all particulars except that one contained iron in the amounts given in the table. The iron salt was made from pure wire, the solution of which was evaporated to dryness several times to remove traces of silica and hydrochloric acid.

The presence of less than 20 parts of iron (Fe) per million of solution introduces no appreciable error in the determination, while as much as 50 parts per million vitiate the phosphate results, unless the amount of iron present is known and its reading subtracted from the total reading. As soil extracts seldom contain more than 0.1 to 5 parts per million of iron, further study of the influence of iron has been temporarily discontinued, but it is hoped to devote more attention to this point later. It is of course necessary, where the presence of considerable iron is indicated, to assure one's self that not more than 20 parts per million are present before the reading can be accepted as representing phosphoric acid alone. This is quite readily ascertained by adding potassium sulphocyanide or ferrocyanide to the acidulated extract, and matching the developed colour against an iron standard (Sutton's "Volumetric Analysis," eighth edition, p. 245).

(To be continued).

TABLE IV.—Colour produced by Iron Salts with Molybdate Solution.

| Parts<br>per million<br>of Fe<br>in solution. | Parts per million of $\text{P}_2\text{O}_5$ to<br>equal colour of Fe solution— |                                                 | Parts per million<br>of $\text{P}_2\text{O}_5$ to equal<br>colour produced by<br>adding 1 part<br>per million $\text{P}_2\text{O}_5$<br>to Fe solution con-<br>taining molybdate. |
|-----------------------------------------------|--------------------------------------------------------------------------------|-------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|                                               | Before<br>molybdate was<br>added.                                              | After molybdate<br>was added to<br>Fe solution. |                                                                                                                                                                                   |
|                                               | <i>Preliminary Series.</i>                                                     |                                                 |                                                                                                                                                                                   |
| 4000                                          | 3.0                                                                            | 6.5                                             | —                                                                                                                                                                                 |
| 2000                                          | —                                                                              | 3.0                                             | —                                                                                                                                                                                 |
| 2000                                          | 0.6                                                                            | 2.5                                             | —                                                                                                                                                                                 |
| 2000                                          | —                                                                              | 4.0                                             | 1.05                                                                                                                                                                              |
| 2000                                          | —                                                                              | 3.1                                             | 0.95                                                                                                                                                                              |
| 2000                                          | —                                                                              | 3.0                                             | 1.10                                                                                                                                                                              |
| 400                                           | 2.2                                                                            | 3.2                                             | —                                                                                                                                                                                 |
| 200                                           | 0.8                                                                            | 1.6                                             | 1.10                                                                                                                                                                              |
| 80                                            | 0.2                                                                            | 0.8                                             | 1.00                                                                                                                                                                              |
| 40                                            | 0.1                                                                            | 0.3                                             | 1.00                                                                                                                                                                              |
| 40                                            | 0.1                                                                            | 0.5                                             | —                                                                                                                                                                                 |
| 40                                            | 0.1                                                                            | 0.5                                             | 1.00                                                                                                                                                                              |
| <i>Final Series.</i>                          |                                                                                |                                                 |                                                                                                                                                                                   |
| 100                                           | 0.1                                                                            | 0.75                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.75                                            |                                                                                                                                                                                   |
|                                               |                                                                                | 0.80                                            |                                                                                                                                                                                   |
| 50                                            | 0.0                                                                            | 0.40                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.40                                            |                                                                                                                                                                                   |
|                                               |                                                                                | 0.50                                            |                                                                                                                                                                                   |
| 20                                            | 0.0                                                                            | 0.10                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.10                                            |                                                                                                                                                                                   |
| 10                                            | 0.0                                                                            | 0.00                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.00                                            |                                                                                                                                                                                   |
| 8                                             | 0.0                                                                            | 0.00                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.00                                            |                                                                                                                                                                                   |
| 4                                             | 0.0                                                                            | 0.00                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.00                                            |                                                                                                                                                                                   |
|                                               |                                                                                | 0.00                                            |                                                                                                                                                                                   |
|                                               | 0.0                                                                            | 0.00                                            | {                                                                                                                                                                                 |
|                                               |                                                                                | 0.00                                            |                                                                                                                                                                                   |

## ON THE PART PLAYED BY BENZENE IN POISONING BY COAL-GAS.\*

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In a recent paper Vahlen† has maintained that a difference exists between the poisonous action of coal-gas and of carbon monoxide, and Kunkel‡ has also drawn attention to a similar difference in the case of frogs. In the course of a research which I was undertaking in University College for other purposes, at the suggestion of Prof. Starling, I have come across facts which may serve to explain the difference noted by these observers.

My first object was to investigate the effect of deprivation of oxygen on the fatigue curve of muscles. To this end, a muscle was hung up in a closed chamber, and the atmospheric air driven out by a stream of some other gas. When coal-gas was used for this purpose, it was noticed that the muscle rapidly went into *rigor mortis*, whereas, in nitrogen, it remained excitable for many hours. I set myself, therefore, to find out which constituent of the coal-gas was responsible for this poisonous effect.

The experiments were carried out on the frog's sartorius, since the relatively large surface of this muscle renders it particularly accessible to gaseous poisons. The muscle was hung up in a glass tube, closed above and below by rubber corks. To the upper cork a hook was fastened on which was hung the bony insertion of the sartorius. Through the lower cork a glass tube passed. The movements of the muscle were transmitted to a recording lever

\* A Paper read before the Royal Society, January 28, 1904.

† Ferchland and Vahlen, "Ueber Verschiedenheit von Leuchtgas- und Kohlenoxydvergiftung," *Archiv für Exper. Pathologie und Pharmacologie* xlviii., p. 106; Vahlen, "Ueber Leuchtgasvergiftung," *Ibid.*, xlix., p. 245.

‡ Kunkel, "Ueber Verschiedenheit von Leuchtgas- und Kohlenoxydvergiftung," *Sitzungsberichte der Physikalisch-medizinischen Gesellschaft zu Würzburg*, 1902, No. 4, v., p. 61.

by means of a steel needle hooked into the lower end of the muscle and passing through the glass tube. The lever was weighted near its axle, and was after-loaded, and its movements were recorded on a slowly rotating drum, on which the time was also marked by means of a signal. The closed tube was provided with two small lateral openings by which any gas desired could be passed through it, the opening through which the needle passed to the lever being made sufficiently air-tight by means of a small plug of vaseline. In every experiment two similar apparatus were employed, so that the influence of two gases could be compared at one and the same time on the two sartorii of the same frog.

When coal-gas was led through the tube the muscle began to contract one to seven minutes after the beginning of the passage of the gas, and in half to one hour the muscle was maximally contracted, and had an opaque appearance. The rapidity with which the contraction came on was proportional to the rapidity with which the gas was passed through the chamber and varied with the temperature, warmth quickening and cold slowing the process. Once the muscle was fully contracted no recovery took place. This phenomenon could not be due to the CO in the coal-gas, since, on passing pure CO through the muscle chamber, the muscle remained excitable as long as in nitrogen, and the fatigue curve took the same course as in that gas. Among the remaining more important constituents of coal-gas which might produce this phenomenon, benzene merited first attention, since all aromatic bodies are more or less potent poisons. I therefore tried the effect of passing air through benzene into the muscle chamber. I found that in less than a minute the muscle began to contract, and the contraction reached its maximum height within a short time, the muscle becoming opaque and dead. The other sartorius of the same frog was placed at the same time in coal-gas. It began to contract only after several minutes, and the contraction took half-an-hour to reach its maximum. Benzene vapour therefore showed itself much more poisonous for a muscle than coal-gas, evidently on account of its greater concentration. According to Letheby, London coal-gas contains only 3.8 per cent of condensable hydrocarbons of which benzene forms only a small proportion. I therefore, in another experiment, passed air into the muscle chamber, not through pure benzene, but through water which had been shaken up with benzene. In this case the contraction began six minutes after the beginning of the experiment, and took thirty-eight minutes to reach its maximum, while the control muscle in coal-gas began to contract in seven minutes, and reached its maximum contraction in forty minutes.

It seemed, therefore, highly probable that benzene was really the constituent of coal-gas which was responsible for its toxic properties on muscle. If that were the case the passage of coal-gas through oil, which absorbs benzene, ought to deprive it of its deleterious effects. This was found to be the case. Coal-gas, passed through oil, showed no difference in its effects from pure CO or nitrogen.

In the same manner as benzene I investigated the effects of xylol and toluol. If the muscle was supplied with air blown through either of these fluids no poisonous effect was observed even when the fluids were warmed. The absence of effect in these two cases is perhaps to be ascribed to the lower vapour tension of these two substances. The muscle was equally unaffected when supplied with air which had been passed over heated naphthalene.

I also investigated the effect of certain hydrocarbons of the fatty series, namely, methane, prepared by heating sodium acetate and sodium hydrate, acetylene produced by the action of water on calcium carbide, as well as the mixture of substances obtained by blowing air through petroleum ether. In none of these cases was any effect produced on the muscle. If, however, a trace of benzene was added to petroleum ether, *rigor mortis* of the muscle was almost immediately produced. In this case the volatility of the petroleum ether apparently aids the evolu-

tion of the benzene. On the other hand, the addition of small quantities of benzene to xylol does not impart poisonous qualities to the air bubbled through the mixture, the benzene being apparently held fast by the less volatile xylol.

It seemed, therefore, most probable that the specific poisonous effect of coal-gas on frog's muscle was due to benzene only, and that it was to the presence of this substance in coal-gas that the differences observed by Kunkel and Vahlen between the action of coal-gas and carbon monoxide were to be referred. Vahlen states that warm-blooded animals and frogs die more rapidly in coal-gas than would be expected from its percentage of CO, and also that frogs in coal-gas present excitatory phenomena which are absent in pure CO. Although Kunkel denies the presence of any difference between the action of these two gases on warm-blooded animals, he also draws attention to the peculiar effects on frogs of coal-gas, which are not produced by other gases free from oxygen, and describes them as "choreiform twitchings and spasms in the neck and legs." To decide this question the following experiments were carried out:—

I. Three frogs were placed in air-tight bell jars, through each of which coal-gas was conducted at constant rate. The coal-gas had to pass in each case through a wash bottle, which in A was water, which, of course, left the composition of the gas unchanged, in B oil, which would absorb the benzene. In front of C were two wash bottles, the first one containing oil, the second one benzene.

The passage of the gas through the three bell jars was begun at 11.20. At 11.25 all three frogs were restless. Frog C remained then still for a short time, and the breathing became irregular, and twitching occurred in the extremities and back. Movements were chiefly co-ordinated, though there were some twitchings of isolated muscles. After a little time the movements became shorter and less co-ordinated, the legs remained stretched out, and breathing ceased. Frog A betrayed phenomena similar to C, but the spasms were less evident, and came on more slowly. Frog B became quite quiet, the respiration becoming irregular and shallower.

At 11.40 B was sitting up in normal position, though the breathing was somewhat irregular, while A and C were lying on their bellies, with legs stretched out. At 11.45 all three frogs were taken out. C gave no signs of life, and in ten minutes was quite rigid; B still reacted slightly to stimulation, showed shallow respiratory movements, and gradually recovered, so that at 1.30 it was apparently normal. Frog A at first showed no response to stimulus, and no respiratory movements. By 11.55 it had recovered sufficiently to show both these phenomena, and at 1.30 it was so far recovered that it could recover its position when turned on its back, and in a couple of hours later was apparently normal.

II. In a second experiment the arrangements were the same as in the first, except that in C the gas which had passed through the oil was allowed to pass through water saturated with benzene instead of through pure benzene. In this case the phenomena in A and C were practically identical, and were similar to those observed in A in the first experiment. We need not, therefore, give fuller details of this experiment.

We thus see that, when frogs are exposed to coal-gas, motor phenomena are produced, which are absent if the coal-gas be previously purified by passage through oil, and that these phenomena can be reproduced if the purified gas be made to take up benzene vapour. The poisonous properties of the gas can be increased by increasing the tension of the benzene vapour. We are therefore justified in concluding that the differences between the effects of CO and coal-gas, observed by Kunkel and Vahlen, depend on the presence in the latter of benzene. The slight motor excitation observed in frogs in coal-gas, which had been purified by passage through oil, is exactly similar

to that described by Kunkel, as the result of deprivation of oxygen, produced by placing the frogs in nitrogen or CO, as is shown by the following experiment:—

III. Two frogs were placed, one in a bell jar through which CO gas was led, the other in a similar jar through which coal-gas was led after passing through oil. Both animals in a short time showed slight twitchings of isolated groups of muscles in the extremities and back, and occasional extensor movements of the hind limbs which gradually diminished. No difference was observable between the two frogs. In three-quarters of an hour they were taken out of the jars, and both recovered within a short time.

In order to be certain of the part played by benzene in coal-gas poisoning, we must have some idea of the effect of pure benzene on the frogs. I have been unable to find any published experiments over the effects of inhalation of benzene on the frog. Beyer\* states that xylol acts as a narcotic poison, like the other odorous substances investigated by him. I have therefore made some experiments on the influence of benzene vapour in the presence of oxygen on frogs.

IV. A frog was placed in a bell jar, in which a beaker of benzene was hung up. Eight minutes after the beginning of the experiment spasmodic movements and twitchings began in various parts of the body, accompanied by a considerable secretion of mucus. After a few minutes the spasms ceased, the frog lay still with extended limbs, and respiration, which at first was irregular, became gradually shallower and less frequent. Twenty minutes after the beginning of the experiment all respiratory and other movements had ceased. The frog was taken out of the jar, and recovered in a few hours.

In other experiments in which the frog was left longer exposed to the action of benzene vapour, *rigor mortis* came on first in the hinder and then in the fore extremities before the heart had ceased to beat. One peculiarity was observed with regard to the reflex irritability of the animals, under these conditions, which is worthy of notice. Very early in the experiment, at the very beginning of the spasmodic movements, the frog reacted very slightly and incompletely to changes in its position, that is, such as would be produced by holding the vessel in which they were placed in an oblique position or turning them on their sides or backs. On the other hand, the reactions to tactile stimuli of the skin were much more pronounced than usual, so that a tap on the one foot might evoke muscular contractions throughout the whole body. When the animals began to become rigid, stimulation of a toe of the rigid limb could evoke contractions of the limbs which had not yet become stiff. Thus, in all these experiments the first result of the poisoning was motor excitation, which showed itself at first by co-ordinated movements affecting large portions of the body, and later by twitchings of isolated groups of muscles. A little later the respiration became irregular, and finally ceased. The higher reflexes, e.g., the reaction to changes in position, were abolished, while the lower were increased. Finally, however, the paralysis became universal, so that also the spinal reflexes were abolished. This stage was followed by a rigidity of the muscles, and last of all the heart ceased to beat. The rapidity of onset of these phenomena is naturally dependent on temperature, being quicker the higher the external temperature. It is evident that we have, therefore, to deal with an action of the poison on the central nervous system. The general spasmodic movements are abolished by previous destruction of the brain and spinal cord; the twitchings of the muscles and the rigor of the extremities persist, however, in the complete absence of the central nervous system, and occur in a hind limb, the nerve of which has been divided, as rapidly as on the opposite side.

The onset is not prevented by curarisation, and must therefore be ascribed to a direct action of the benzene on the muscles themselves, as has been described at the beginning of this paper.

In the poisoning by coal-gas this rigor of the muscles was not observed either by myself or Vahlen and Kunkel, probably because the animals die of asphyxia before the small amounts of benzene present in the coal-gas have time to bring about their direct effect upon the muscular tissue. As Experiment I. shows, the increase in the percentage of benzene in the coal-gas is followed by the onset of rigidity in the muscles.

Rather more difficult is the explanation of the increased reflex excitability in the later stages of intoxication. It may be that the poisonous effects are first confined to the higher centres. On the other hand, the increased irritability of the muscles themselves is the chief factor in the spinal reflex hyper-excitability. In coal-gas poison, when the lower centres are also paralysed by asphyxia, the reflex excitability disappears.

It is thus possible to refer the difference between intoxication by coal-gas and that by CO entirely to the influence of benzene, which determines in its first stage vigorous excitatory motor phenomena. In warm-blooded animals the conditions are quite different. Lorraine Smith\* found that an addition of 0.65 percent of benzene to air had no effects on a guinea-pig, and Santesson† did not succeed in producing either acute or chronic poisonous effects by administering benzene to a rabbit by inhalation. In man, too, cases of poisoning by benzene are few and far between. It is possible that the minute quantities which are absorbed by the lungs are rapidly oxidised and excreted as an aromatic sulphate. At any rate, in man, benzene plays no part in the poisonous effects of coal-gas.

#### Summary of Results.

1. Coal-gas produces first excitation and then rigor of the isolated frog's muscle.
2. Frogs exposed to coal-gas show excitatory phenomena which are absent when the animal is placed in an atmosphere of CO or nitrogen.
3. The specific effects of coal-gas on frogs are determined by the presence of benzene in the gas, and can be produced by air containing the same percentage of benzene.
4. There is no reason to suppose that the poisonous effect of coal-gas on mammals is determined by anything except its content in CO.

### PRECIPITATION AND SEPARATION BY WEAK ORGANIC BASES.‡

By E. T. ALLEN.

(Continued from p. 65).

#### Separation of Titanium from Beryllium.

It has been shown that the elements of the quadrivalent family, Ti—Th, are, with the exception of Ce, separable from iron by phenylhydrazine. Beryllium forms a much weaker base than ferrous iron, but it may be separated from titanium and zirconium in a fairly satisfactory manner by similar means. The tendency is for the beryllium also to be partly precipitated, and with sulphate solutions, especially where the quantities of the elements to be separated are considerable, satisfactory results are not obtained by two precipitations. The standard solution of beryllium employed in the work was the chloride. The source of it

\* Beyer, "Narkotische Wirkungen von Riechstoffen und ihr Einfluss auf die Motorischen Nerven des Frosches," *Archiv für Anatomie und Physiologie, Ph; Biologische Abteilung*, 1902, p. 201.

\* Lorraine Smith, "The Poisonous Action of Coal-gas and Carburetted Water-gas." "Report of the Water-gas Committee," presented to both Houses of Parliament by command of Her Majesty, 1899, Appendix VII., p. 127.

† Santesson, "Ueber Chronische Vergiftungen mit Steinkohlenbenzin," *Archiv für Hygiene*, xxxi., p. 336.

‡ From the *Journal of the American Chemical Society*, xxv., No. 4.

TABLE D.—Separation of Titanium from Beryllium (Double Precipitation).

| No. | BeCl <sub>2</sub> taken.<br>C.c. | BeO.<br>Grm. | BeO found.<br>Grm. | Error.<br>Grm. | Ti(SO <sub>4</sub> ) <sub>2</sub> taken.<br>C.c. | TiO <sub>2</sub> .<br>Grm. | TiO <sub>2</sub> found.<br>Grm. | Error.<br>Grm. |
|-----|----------------------------------|--------------|--------------------|----------------|--------------------------------------------------|----------------------------|---------------------------------|----------------|
| 1.  | 10                               | =            | 0.0101             | —              | 10                                               | =                          | 0.0096                          | 0.0000         |
| 2.  | 10                               | =            | 0.0101             | —              | 10                                               | =                          | 0.0096                          | 0.0000         |
| 3.  | 5                                | =            | 0.0050             | —              | 25                                               | =                          | 0.0239                          | -0.0008        |
| 4.  | 30                               | =            | 0.0302             | —              | 5                                                | =                          | 0.0048                          | +0.0021        |
| 5.  | 100                              | =            | 0.1006             | 0.0960         | 100                                              | =                          | 0.0958                          | +0.0038        |
| 6.  | 95                               | =            | 0.0956             | 0.0951         | 5                                                | =                          | 0.0048                          | +0.0006        |
| 7.  | 5                                | =            | 0.0050             | 0.0049         | 95                                               | =                          | 0.0910                          | +0.0001        |

TABLE E.

| No. | BeCl <sub>2</sub> taken.<br>C.c. | BeO.<br>Grm. | BeO found.<br>Grm. | Error.<br>Grm. | Zr(SO <sub>4</sub> ) <sub>2</sub> taken.<br>C.c. | ZrO <sub>2</sub> .<br>Grm. | ZrO <sub>2</sub> found.<br>Grm. | Error.<br>Grm. |
|-----|----------------------------------|--------------|--------------------|----------------|--------------------------------------------------|----------------------------|---------------------------------|----------------|
| 1.  | 5                                | =            | 0.0050             | —              | 95                                               | =                          | 0.0925                          | +0.0036        |
| 2.  | 95                               | =            | 0.0956             | —              | 5                                                | =                          | 0.0049                          | +0.0012        |
| 3.  | 5                                | =            | 0.0066             | 0.0040         | 45                                               | =                          | 0.0438                          | +0.0020        |
| 4.  | 45                               | =            | 0.0591             | 0.0590         | 5                                                | =                          | 0.0049                          | +0.0003        |
| 5.  | 15                               | =            | 0.0196             | 0.0187         | 45                                               | =                          | 0.0438                          | +0.0005        |
| 6.  | 35                               | =            | 0.0458             | 0.0445         | 35                                               | =                          | 0.0341                          | +0.0018        |
| 7.  | 100                              | =            | 0.1305             | 0.1290         | 50                                               | =                          | 0.0506                          | +0.0014        |

was beryl from Chester County, Pennsylvania. I transformed this into the double fluoride by the method which Margnac used in extracting zirconia from zircon, viz., by fusing the powdered mineral with two or three times its weight of potassium fluoride. For convenience in handling, the fused mass may be poured out in a thin sheet into a platinum basin and subsequently powdered. It is then boiled several times with successive portions of water, to which should be added at first some hydrofluoric acid. The double fluorides which silicon and aluminum form with potassium are nearly insoluble in water containing potassium fluoride, and are thus for the most part left behind. The solution is now evaporated to a small volume and allowed to crystallise. The salt is then re-crystallised several times. It dissolves rather slowly in boiling water. Each time the last portion should be rejected. The compound still retains iron, which may be entirely removed by passing hydrogen sulphide through the aqueous solution, which is, of course, alkaline. The crystals may finally be dried at 105° C. It is a difficult matter to be sure of the complete absence of alumina, but the following determination indicates that the compound thus prepared is essentially pure. A weighed portion was changed into sulphate by heating with sulphuric acid, thence through the hydroxide to chloride, and finally to hydroxide again, which was ignited and weighed. To be sure that the beryllium oxide was free from sulphate, the precipitation with ammonia was, in fact, made three times. All the filtrates were evaporated to a small volume and treated with a little ammonia, which brought down a little beryllia, which was filtered and washed separately and added to the main portion. 1.2996 grms. gave 0.2008 grm. BeO; calculated for K<sub>2</sub>BeF<sub>4</sub>, 0.1993. I am not aware that this method has been used previously to extract beryllium from beryl. For the determinations which follow, a solution of beryllium chloride was prepared as above indicated. To separate titanium and zirconium from beryllium, it is not, of course, necessary to add any soluble sulphite. The acid solutions are merely brought nearly to neutrality with ammonia and then acidified with several drops of dilute hydrochloric acid. A double precipitation was made, and water only was used for washing. The solutions were heated but not boiled. As previously stated, the results were not so exact as in the separation of titanium and zirconium from ferrous iron.

The beryllium was determined in the filtrate from titanium by ammonia, as in an ordinary determination of aluminum. Subsequent experiments showed that phenylhydrazine precipitates a large fraction of the beryllium from a solution of the sulphate, but none from the chloride. It follows that a double precipitation by phenylhydrazine from a chloride solution would give a more exact separation from titanium than those recorded in Table D.

#### Separation of Titanium from Beryllium by Aniline.

| No. | BeO taken.<br>Grm. | TiO <sub>2</sub> taken.<br>Grm. | TiO <sub>2</sub> found.<br>Grm. | Error.<br>Grm. |
|-----|--------------------|---------------------------------|---------------------------------|----------------|
| 1.  | 0.050              | 0.1416                          | 0.1422                          | -0.0006        |
| 2.  | 0.075              | 0.0944                          | 0.0950                          | -0.0006        |

The solutions used were beryllium chloride and titanium sulphate. The mixtures were first changed to chlorides by precipitation with ammonia and subsequent solution in hydrochloric acid. From the chloride solution, a double precipitation was made by aniline.

#### Separation of Zirconium from Beryllium.

Everything that has been said as regards the separation of titanium from beryllium applies equally well here. (See Table E).

An inspection of the tables makes it plain that a little beryllium is sure to be carried down with the quadrivalent metal where sulphate solutions are used, but here too, as in the case of beryllium and titanium, we may confidently predict more accurate results if chloride solutions are taken.

#### Separation of Thorium from Beryllium.

| No. | BeO taken.<br>Grm. | ThO <sub>2</sub> taken.<br>Grm. | ThO <sub>2</sub> found.<br>Grm. | Error.<br>Grm. |
|-----|--------------------|---------------------------------|---------------------------------|----------------|
| 1.  | 0.025              | 0.2863                          | 0.2847                          | -0.0016        |
| 2.  | 0.025              | 0.1909                          | 0.1895                          | -0.0014        |

The solutions used were beryllium chloride and thorium nitrate. Separation 1 was made with phenylhydrazine, separation 2 with aniline. Double precipitations were made in both cases. Thorium is, of course, a stronger base than titania or zirconia. From the above solutions it did not precipitate until a boiling temperature was reached and the precipitate was very slimy, like alumina when precipitated under the same circumstances. This may account for the fact that the results are a little low.

Attempts to separate aluminum from beryllium, and chromium from beryllium, and from ferrous iron, all failed. Usually the bivalent metal was carried down in considerable quantities with the trivalent one. Chromium chloride does not easily precipitate with phenylhydrazine. Many experiments tried along the above lines show that two elements when too near the same basicity can not be separated by this method, though just why the bivalent element should be partly precipitated in the presence of the trivalent is not perfectly clear.

#### The Hydrolysis of Aniline and Phenylhydrazine Hydrochlorides.

The degree of hydrolysis which a salt suffers in aqueous solution may be estimated from its conductivity, the values observed being compared with those shown by salts

of the same type in which hydrolysis is negligible; or it may be calculated from the velocity of certain chemical actions which are caused by the free acid present in the salt solution. The best examples of the latter are the inversion of cane-sugar, and the saponification of methyl acetate. The last-named method was selected in this work (Walker, *Zeit. Phys. Chim.*, iv., 319). A medium temperature of 40° C. was chosen. It was maintained by thermostat of the Ostwald type, of 100 litres capacity, which could be held within limits of about 0.2° C. for long periods. All the experiments were made with N/10 solutions of the hydrochlorides of the two bases, phenylhydrazine and aniline. Several preparations were used that there might be reasonable certainty that the saponification constants were not affected in any considerable degree by impurities. Two different samples of "C.P." phenylhydrazine hydrochloride were purified by dissolving in alcohol and precipitating by ether. One specimen of aniline hydrochloride was prepared in a similar way, and another was made from pure aniline by evaporating with pure hydrochloric acid on the water-bath. All the preparations stood a long time in desiccators with lime and sulphuric acid, before use. The reaction vessels consisted of 8-inch test-tubes which were first treated for some time with steam, then boiled with concentrated hydrochloric acid, and finally very thoroughly washed and dried (Ley, *Zeit. Phys. Chem.*, xxx., 229; Walker, *Journ. Chem. Soc.*, lxxv., 577). N/10 solutions of the hydrochlorides were employed in the saponification as representing on the average about the concentration of these salts in those solutions in which the separations were made. In detail, the proper quantities of the salts, viz., 0.324 gm.  $C_6H_5.NH_2Cl$ , and 0.361 gm.  $C_6H_5.N_2H_4Cl$ , were first weighed out and transferred to the reaction tubes. The latter were then constricted in the flame at a distance of 2 or 3 inches from the open end, after which 1 c.c. methyl acetate was introduced by a pipette about 2 m.m. in diameter, followed by 250 c.c. water free from carbonic acid. The tubes were then sealed, cooled, shaken to mix the contents, and placed in the bath. After a sufficient interval, a tube was withdrawn, chilled, and opened. Its contents were then tested as follows:—1 c.c. was removed by a pipette, added to a small volume of water free from carbonic acid, and titrated with N/20 soda, phenolphthalein serving as an indicator. The errors in measurement were reduced to a minimum by using narrow instruments. The pipette was about 2 m.m. in diameter and the 10 c.c. burette, about 8 m.m. During the titration, the contents of the burette were protected from the outer air by a soda-lime tube. The sodium hydroxide was prepared according to Ley (*Zeit. Phys. Chem.*, xxx., 205), and was proved free from carbon dioxide by titrating against N/10 hydrochloric acid, first with the aid of phenolphthalein, then with methyl orange.

1. Phenolphthalein used as indicator:—5 c.c. HCl = 9.58, 9.59, 9.58, 9.60 c.c. NaOH; mean, 9.59 c.c.
2. Methyl orange used as an indicator:—5 c.c. HCl = 9.59, 9.52, 9.58, 9.59 c.c. NaOH; mean, 9.57 c.c.

(To be continued).

**Analysis of the Combustible Gas given off in the Caspian Sea, near the Gulf of Baku.**—K. V. Charitchkof. —Taking the sample is a matter of some difficulty, and it is almost impossible to obtain the gas free from all admixture with atmospheric air; we have taken account, in the analysis, of this air, calculated on the quantity of oxygen measured. The gases, collected in two different places, are shown to be free from  $CO_2$ , CO, and non-saturated hydrocarbons, neither do they contain hydrogen (tested for with palladium sponge by Hempel's method); they consist of methane and a little nitrogen:—

|                         | First gas. | Second gas. |
|-------------------------|------------|-------------|
| $CH_4$ , per cent .. .. | 96.28      | 95.17       |
| N, per cent .. ..       | 3.72       | 4.83        |

—*Journ. Soc. Phys. Chim. R.*, vol. xxxiv., p. 712.

## INSTITUTE OF CHEMISTRY.

At the January Examinations, 1904, the following questions were given:—

### INTERMEDIATE EXAMINATION.

#### General and Theoretical Chemistry.

Tuesday, January 5th.

Not more than four of the following questions are to be attempted:—

1. How would you prepare silicon hydride, silicon fluoride, and an aqueous solution of silicic acid? What are the properties of these substances? What reasons exist for classifying silicon with carbon and tin.
2. Give an account of the oxides of lead, and explain the use of lead peroxide in the storage battery or accumulator.
3. How would you proceed to determine the composition of a sample of gas obtained by the action of a mixture of steam and air on red-hot coke?
4. Ethylene and ethylidene chlorides are isomeric substances. What is the evidence on which this statement is based, and how can the constitution of the chlorides be determined?
5. Describe fully the preparation in the pure state, the properties, and the reactions of any two of the following substances:—Allyl alcohol, lactic acid, acetophenone, resorcinol.
6. Explain the methods by which salicylic acid can be prepared synthetically. How may this acid be proved to be an ortho-derivative of benzene, and how may it be distinguished from its isomerides? How could it be converted into (a) benzene, (b) phenol, and (c) benzoic acid?

Not more than four of the following questions are to be attempted:—

7. Write an essay on nitrogen peroxide.
8. Describe the preparation in the pure state, appearance, and properties of two of the following substances:—Phosphonium iodide, hydroxylamine sulphate, sodium metabisulphite; potassium persulphate, anhydrous chromium chloride.
9. What is iron rust? What views are held with regard to the chemistry of the process of rusting? How do you account for the fact that tinned iron rusts more rapidly than galvanised iron when once the oxidation has begun?
10. What conclusions do you draw from the following results obtained in the course of the investigation of an ester:—(a) Found, C = 46.32, H = 7.16, O (by diff.) = 46.52 per cent; (b) 1.5225 grms. dissolved in 33.58 grms. of ethyl alcohol raised the boiling-point by 0.245° (constant = 11.7); (c) a 10.94 per cent solution in ethyl alcohol at 18.6° gave  $n_D + 3.023$  in a 400 m.m. tube (sp. gr. of solution 0.8251).
11. From what sources are glucose and levulose prepared, and in what respects do they differ from one another? Give the constitutional formulæ of these sugars, and describe how one of them can be converted into the other.
12. Give an account of the reactions which nitrous acid yields with organic compounds. How would you ascertain whether a compound containing nitrogen and oxygen were a nitroso-derivative or not?

### Practical Chemistry.

Wednesday, January 6th.

1. A is a solution of zinc and magnesium sulphates in water. Ascertain the amount of each in one litre of the solution.
2. Determine the melting-point of B (benzamide). To what type of compound does this substance belong?

Thursday, January 7th.

A. Analyse qualitatively the given mixture. (Potassium tartrate, barium carbonate, magnesium phosphate; ammonium oxalate, cadmium carbonate, ammonium magnesium arsenate; sodium salicylate, aluminium phosphate calcium arsenate. One mixture given to each candidate)

B. Determine the percentage of potassium nitrate in the given solution by a gasometric method.

Friday, January 8th.

A is a solution of ammonium chloride and sulphuric acid in water. By means of the given semi-normal solution of sodium hydroxide determine the amount of ammonium chloride present. No other volumetric solution may be used.

B. The given mixture contains two organic substances. (Aniline hydrochloride and  $\alpha$ -naphthol; aniline sulphate and  $\beta$ -naphthol. One mixture given to each candidate). Separate and identify them. Estimate their relative proportion in the mixture and prepare from each a characteristic derivative.

FINAL EXAMINATIONS FOR THE ASSOCIATESHIP.

Branch A.—Mineral Chemistry.

Tuesday, January 12th.

Make a qualitative and quantitative analysis of the mixture A (lead oxalate, lead carbonate, and aluminium phosphate).

The estimation of one metal and of at least one acid radicle should be finished to-day, and the exercise completed to-morrow.

Wednesday, January 13th.

1. Complete the quantitative analysis of the mixture A given yesterday.
2. Ascertain what substances are present in the mixture B (ammonium persulphate and barium peroxide).

Thursday, January 14th.

1. Make an analysis of the sample of "furnace gas," and estimate the amount of each constituent.
2. Examine the given mineral C by the spectroscope and state what elements can be recognised in it (lepidolite).

Friday, January 15th.

1. The substance D is potassium nitrate with an admixture of a sulphite and sulphate. Find the proportion of potassium nitrate and estimate the amount of sulphite present.
2. Make a qualitative analysis of the mineral C examined spectroscopically yesterday.

Branch D.—Organic Chemistry.

Tuesday, January 12th.

1. What is the percentage of anthracene in the given sample of the crude hydrocarbon A? (This exercise may be completed to-morrow).
2. Identify the given substance B (benzenesulphonic chloride), checking your results by the quantitative determination of one constituent. Prepare specimens illustrative of the use to which the substance may be put in the laboratory.

Wednesday, January 13th.

The given liquid is a mixture of two substances (acetone and methyl alcohol). Identify them, determine their proportion in the mixture, and isolate one of them in a pure state. Leave specimens of any derivatives you are able to prepare.

Thursday, January 14th.

Prepare a litre of 3 per cent starch paste. Cool to  $65^{\circ}\text{C}$ ., then add 25 c.c. of the given solution of malt diastase which has been previously raised to  $65^{\circ}\text{C}$ . Maintain the solution at this temperature and determine the reducing power, calculated as maltose, at stated intervals by means of a volumetric process. Plot your results on the squared paper supplied. Quantities of 50 c.c. should be withdrawn at intervals of 2, 4, 7, 10, 15, 30, 60, and 90 minutes, and

the diastatic action in each stopped by the addition of alkali.

Friday, January 15th.

1. The substance supplied is a commercial specimen of a base used in the manufacture of colouring matters (dimethylaniline containing monomethylaniline). Examine it, determine its nature, and report on its purity. Leave specimens of any derivatives you are able to prepare.

2. What do you consider to be the nature of the substance B? (Martius' yellow).

Branch E.—The Analysis of Foods and Drugs, and of Water.

Tuesday, January 5th.

Analyse the condensed milk, estimating the water, fat, proteids, and ash. From your results calculate the ratio between the original milk and the condensed product; state whether the sample is "skimmed."

Wednesday, January 6th.

1. Examine the honey by the polarimeter, and give an opinion as to whether the sample is adulterated with foreign sugars.

2. Find what ingredients have been added to the "self-raising" flour, and determine their amount.

Thursday, January 7th.

1. Identify the alkaloid in the given tincture (belladonna); estimate the quantity present, and state whether the tincture conforms to the requirements of the British Pharmacopœia.

2. Test the given drugs for impurities and determine the proportion of any foreign ingredients. (Two samples given to each candidate).

Friday, January 8th.

1. Examine the water and report on its suitability for use in steam boilers. Give your opinion on the possibility of improving the water and state what treatment you recommend.

2. Write reports on the given samples. (Vinegar, lime-juice, and ginger wine. Two to each candidate).

Candidates in Branch E of the Final Examinations were required to pass an Examination in Therapeutics, Pharmacology, and Microscopy.

Saturday, January 9th.

1. Examine the specimen of mustard marked A, and report on its probable purity or otherwise, basing your opinion on the microscopical appearances only. Leave a prepared slide on your bench.

2. Examine by means of the microscope the specimen marked B. Describe in detail its nature as seen under the microscope. Leave a prepared slide on your bench.

3. Examine, describe, and state respectively the nature of the prepared slides marked C and D.

4. Mention the various metallic impurities that may be present in drinking waters. Describe the sources from which they are respectively derived, and briefly discuss the injurious effects that may be produced on the health of persons drinking such impure waters.

5. Give the full medicinal and the average fatal doses for an adult of the following substances:—Hydrocyanic acid (B.P.), phosphorus, acetanilide, chloral hydrate, croton oil, nitro-glycerin.

Candidates were also examined practically and interrogated orally as to the recognition of chemicals and drugs.

*Examiners in Chemistry*—Walter William Fisher, M.A. (Oxon.), F.I.C.; William Palmer Wynne, D.Sc. (Lond.), F.R.S., F.I.C.

*Examiner in Therapeutics, Pharmacology, and Microscopy*—Arthur Pearson Luff, M.D., B.Sc. (Lond.), F.R.C.P., F.I.C.



## PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, January 20th, 1904.

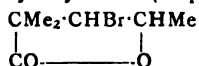
Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

(Concluded from p. 70).

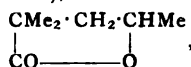
7. "The *cis*- and *trans*-Modifications of  $\alpha\gamma$ -Trimethylglutaconic Acid." By WILLIAM HENRY PERKIN, jun., and ALICE EMILY SMITH.

The authors have already shown (*Trans.*, 1903, lxxxiii., 772) that when ethyl  $\alpha\gamma$ -trimethylacetonedicarboxylate,  $\text{CO}_2\text{Et}\cdot\text{CMe}_2\cdot\text{CO}\cdot\text{CHMe}\cdot\text{CO}_2\text{Et}$ , is reduced with sodium amalgam, it is converted into a mixture of the *cis*- and *trans*-modifications of  $\beta$ -hydroxy- $\alpha\gamma$ -trimethylglutaric acid,  $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}(\text{OH})\cdot\text{CHMe}\cdot\text{CO}_2\text{H}$ , and both of these, by treatment first with phosphorus pentachloride and then with diethylaniline, yield *trans*- $\alpha\gamma$ -trimethylglutaconic acid,  $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}:\text{CMe}:\text{CO}_2\text{H}$ , which melts at  $150^\circ$ . They now show that on distillation both of the foregoing hydroxy-acids are converted into the anhydride of the *cis*-modification of  $\alpha\gamma$ -trimethylglutaconic acid, which melts at  $88^\circ$ , and on hydrolysis yields the corresponding acid melting at about  $125^\circ$ . On treatment with bromine, *cis*-trimethylglutaconic acid yields *cis*- $\gamma$ -dibromo- $\alpha\gamma$ -trimethylglutaric acid,  $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CHBr}\cdot\text{CMeBr}\cdot\text{CO}_2\text{H}$ , which melts at  $168^\circ$ .

During the distillation of the hydroxy-acids, a considerable amount of carbon dioxide and steam is eliminated, and an oily acid is produced which distils at  $213^\circ$ . This substance, *crotonyldimethylacetic acid*,  $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}:\text{CHMe}$ , when treated with bromine, yields the lactone of  $\beta$ -bromo- $\gamma$ -hydroxy- $\alpha\gamma$ -trimethylbutyric acid (m. p.  $83^\circ$ ),—



and, when boiled with dilute sulphuric acid, is converted into the lactone of  $\gamma$ -hydroxy- $\alpha$ - $\gamma$ -trimethylbutyric acid (i. dimethylvalerolactone),—



which melts at  $52^\circ$ , and has already been described by Anschütz and Gillet (*Annalen*, 1888, ccxlvii., 107), who obtained it by the reduction of  $\alpha\alpha$ -dimethylævulic acid,  $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}_2\cdot\text{COMe}$ .

8. "The Influence of Substitution in the Nucleus on the rate of Oxidation of the Side-chain. I. Oxidation of the Mono- and Dichlorotoluenes." By JULIUS BEREND COHEN and JAMES MILLER.

The method of oxidation adopted was to heat about a gram. of each of the isomerides with dilute nitric acid in a sealed tube for a length of time insufficient for complete oxidation and to estimate the proportion of the acid product and the unchanged substance. The vessel employed as air-bath was a jacketed, tin-plate cylinder containing boiling coal-tar naphtha, and furnished with a condenser, the tubes being heated in the inner compartment, which was kept at a nearly constant temperature.

The results of several series of experiments show that the monohalogen derivatives are more rapidly attacked than the dihalogen compounds. Of the three monochlorotoluenes, the meta-compound is least rapidly, and the para-isomeride most rapidly oxidised. Of the dihalogen compounds, the 3:5-compound is least affected; then follow the 2:5- and 2:6-isomerides, which are oxidised about equally; then the 2:3-compounds, and finally the 2:4- and 3:4-compounds, which may be bracketed together as being most readily attacked.

So far as these experiments are concerned, in which the oxidising agent is nitric acid, the results are perfectly

definite. The meta-compounds retard and the para-derivatives assist oxidation, whilst the ortho-compounds occupy an intermediate position.

Thus, *m*-chlorotoluene and 3:5-dichlorotoluene are least attacked by the acid, whereas *p*-chlorotoluene and the 2:4- and 3:4-dichlorotoluenes, which each contain a chlorine atom in the para-position with respect to the methyl group, are most rapidly oxidised.

9. "The Interdependence of the Physical and Chemical Criteria in the Analysis of Butter Fat." By THOMAS EDWARD THORPE.

In the course of an investigation on the chemical nature of butter produced within the British Isles, which was instituted by the Board of Agriculture for the information of a Departmental Committee appointed to report as to what regulations might with advantage be made for determining what deficiency in any of the normal constituents of butter should raise a presumption under the Food and Drugs Acts that the butter was not genuine, it became necessary to obtain at first-hand the values of butter of known origin and produced from milk given under varying conditions. Observation has shown that the chemical nature of butter fat is dependent, to a certain extent, on the climatic influences to which the cows are exposed, on the nature and amount of the food supplied, and on the breed, period of lactation, and iodiosyncrasy of the individual cow. In order to give such weight as was practicable to the effect of these factors, the samples of butter were obtained from carefully selected districts, and often from cows set apart for the purpose of the inquiry, whilst particulars of the breed, diet, stabling, and period of lactation were supplied with the samples in nearly all cases. For example, to illustrate the effects of more rigorous climatic conditions than obtained in the United Kingdom generally, farms and dairies in Caithness, Sutherland, the Orkneys, Shetlands, and the Hebrides were laid under contribution, whilst a series of samples from Hollesley Bay, Suffolk, served to exemplify the effect on the cattle of exposure to the winds of the North Sea.

Of the 430 samples received, 357 were examined as regards their Reichert-Wollny number, their relative density, saponification value, and refractometric value, and in a certain number of typical cases their Hübl value was ascertained.

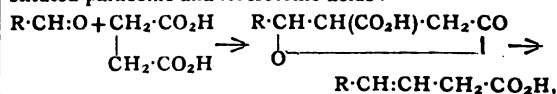
The main results of these observations have been tabulated and compared, and their interdependence exhibited by means of curves.

10. "A simple Thermostat for Use in Connection with the Refractometric Examination of Oils and Fats." By THOMAS EDWARD THORPE.

The thermostat is primarily intended for use in connection with the Zeiss butyro-refractometer, in which a current of water, usually at about  $45^\circ$  or a little warmer, is caused to circulate round the prism casing. This arrangement is somewhat more convenient than that usually employed, occupies less space, and is sooner brought into action. It has for some years past been employed in the Government Laboratory in the examination of butter.

11. "The Condensation of Furfuraldehyde with Sodium Succinate." By ARTHUR WALSH TITHERLEY and JAMES FREDERICK SPENCER.

Fittig has shown (*Annalen*, 1883, ccxvi., 97; 1889, cclv., 1—142) that aldehydes condense with sodium succinate in the presence of acetic anhydride, giving substituted paraconic and isocrotonic acids:—



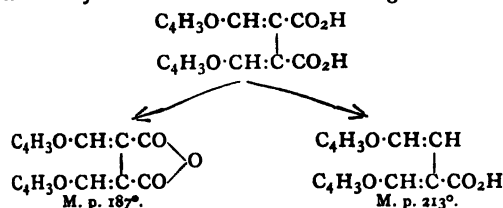
where R is any alkyl or aryl radicle.

The authors in attempting to employ this method for the synthesis of  $\beta$  furfurylidenepropionic (furfurylisocrotonic) acid by condensing furfuraldehyde with sodium succinate in presence of acetic anhydride, found that neither it nor

furfurylparaconic acid is formed, but two unexpected derivatives were produced, which were isolated with considerable difficulty, the yields being very small.

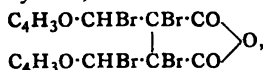
One of these substances, which crystallised in dark orange-coloured needles melting at  $187^{\circ}$ , was found to be *difurfurylidene-succinic anhydride*,  $C_{14}H_8O_5$ , and the other, which separated in bright yellow plates (m. p.  $213^{\circ}$ ), was identified as  $\alpha\gamma$ -difurfurylidene-*propionic acid*,  $C_{13}H_{10}O_4$ . These results indicated that two furfuraldehyde molecules condensed with one molecule of sodium succinate, and all attempts to prepare the corresponding monofurfurylidene derivatives failed.

These products may be regarded as being produced from the difurfurylidene derivative in the following manner:—



The orange-coloured anhydride is decomposed with difficulty by sodium hydroxide, forming sodium *difurfurylidene-succinate*; the acid is a yellow, crystalline powder melting at  $185$ – $187^{\circ}$ , and regenerating the anhydride. The acid is also converted into the anhydride on gently warming with acetyl chloride.

The anhydride readily combines with bromine, yielding the well-defined tetrabromide,  $\alpha\beta\gamma\delta$ -tetrabromo- $\alpha\gamma$ -difurfurylsuccinic anhydride,—



a bright yellow powder melting at  $196^{\circ}$ , and giving fluorescent solutions.

The yellow  $\alpha\gamma$ -difurfurylidene-*propionic acid* (m. p.  $213^{\circ}$ ) was obtained as the sole crystallisable product and in larger yield, on using succinic anhydride as the dehydrating agent instead of acetic anhydride, but in all cases the condensation of furfuraldehyde with sodium succinate was accompanied by the formation of large quantities of resinous matter.

12. "The Action of Heat on  $\alpha$ -Hydroxycarboxylic Acids." (A Preliminary Note). By HENRY RONDEL LE SUEUR.

The aldehyde,  $C_{16}H_{33}CHO$ , which is produced by heating  $\alpha$ -hydroxystearic acid, crystallises from light petroleum in needles melting at  $36^{\circ}$ , and from alcohol in needles containing 1 mol. of the solvent, and melting at  $52^{\circ}$ ; it forms a crystalline compound with sodium hydrogen sulphite, and the oxime and semicarbazone melt at  $89.5^{\circ}$  and  $107$ – $108^{\circ}$  respectively.

The acid,  $C_{16}H_{33}CO_2H$ , obtained by oxidising the aldehyde with potassium permanganate, crystallises from light petroleum in long needles melting at  $60$ – $61^{\circ}$ ; the silver salt,  $C_{17}H_{33}O_2Ag$ , is amorphous.

The author is investigating the action of heat on other mono- and di-basic  $\alpha$ -hydroxy-acids.

13. "The Fusion of Isopilocarpine with Caustic Potash." (A Correction). By HOOPER ALBERT DICKINSON JOWETT.

In a communication on the constitution of pilocarpine (*Trans.*, 1900, lxxvii., 860), the author showed that an acid, then regarded as isobutyric acid, was produced by the fusion of isopilocarpine with caustic potash. The existence of the *n*-butyl group in isopilocarpine, as proved by the formation of  $\alpha$ -ethyltricarballic acid from homopilocarpic acid, and the production of *n*-butyric acid by fusion of pilocarpic acid with caustic potash, however, rendered it probable that the acid previously described as isobutyric acid was really *n*-butyric acid. The experiment has therefore been repeated.

Ten grms. of isopilocarpine were fused with 100 grms. of caustic potash, and the fatty acid separated in the manner previously described (*loc. cit.*). The portion distilling between  $120^{\circ}$  and  $160^{\circ}$  was collected and converted into the calcium salt by neutralisation with calcium carbonate. The acid was not soluble in a small amount of water, but dissolved almost completely in a larger quantity. The calcium salt, which separated from the hot aqueous solution on concentration, was collected while hot; it formed white, pearly plates, which were dried in the air and analysed:—

$0.1982$  lost  $0.0162$   $H_2O$  at  $150^{\circ}$ .  $H_2O = 8.2$ .

$0.1780$  lost  $0.0144$   $H_2O$  at  $100^{\circ}$ .  $H_2O = 8.1$ .

$(C_4H_7O_2)_2Ca \cdot H_2O$  requires  $H_2O = 7.8$  per cent.

Calcium isobutyrate crystallises with 4 molecules of water.

On warming an aqueous solution of the salt, saturated at  $0^{\circ}$ , crystals separated, which re-dissolved on cooling.

The silver salt was prepared and re-crystallised from water.

$0.1312$  gave  $0.0726$  Ag. Ag =  $55.3$ .

$C_4H_7O_2Ag$  requires Ag =  $55.4$  per cent.

The volatile acid formed by the fusion of isopilocarpine with caustic potash is therefore *n*-butyric acid.

14. "Organic Derivatives of Silicon. Preparation of Alkylsilicon Chlorides." By FREDERIC STANLEY KIPPING.

It has been previously shown that tetra-alkyl derivatives of silicon can be obtained by treating silicon tetrachloride with alkyl halides in presence of sodium (Kipping and Lloyd (*Trans.*, 1901, lxxix., 449)). The investigation of silicon compounds has recently been resumed in conjunction with Mr. A. Hunter, and attempts have been made to prepare compounds of the type  $SiR_1R_2R_3Cl$  with the following results:—

Silicon tetrachloride interacts vigorously with an ethereal solution of magnesium ethyl iodide, the product consisting of a mixture of ethylsilicon trichloride, diethylsilicon dichloride, triethylsilicon chloride, and silicon tetraethyl, the proportions of which depend on the relative quantity of the magnesium compound employed.

Silicon tetrachloride also interacts very vigorously with an ethereal solution of magnesium ethyl bromide; when molecular proportions of the two compounds are used, the product contains a small quantity of the di- and tri-ethyl derivatives, but consists principally of ethyl silicon trichloride, which can be isolated by fractional distillation and has the properties assigned to it by Ladenburg.

Ethylsilicon trichloride and an ethereal solution of magnesium phenyl bromide interact very readily, and phenyl-ethylsilicon dichloride can be isolated by fractional distillation; this compound is a fuming liquid boiling at about  $228$ – $230^{\circ}$ , and readily decomposed by water, giving an oil which appears to be the *silicoketone*,  $SiEtPhO$ .

Phenylethylsilicon dichloride and an ethereal solution of magnesium propyl bromide do not give an immediate precipitation of magnesium salt, but on warming, a reaction occurs with the formation of phenylethylpropylsilicon chloride, which is obtained after fractional distillation, mixed apparently with unchanged dichloride, as a colourless liquid boiling indefinitely at about  $240^{\circ}$ .

These and other alkyl derivatives of silicon tetrachloride are now being examined, and, in conjunction with Dr. Caven, analogous experiments have been commenced with the object of preparing mixed alkyl derivatives of phosphorus, more especially compounds of the type  $POR_1R_2R_3$  or  $POR_1R_2OH$ .

15. "Derivatives of Highly Substituted Anilines." By FREDERICK DANIEL CHATTAWAY and JOHN MELLO WADMORE.

The authors described the preparation and properties of a series of highly substituted acyl- and chloro-amines obtained in the course of an investigation into the phenomena of intramolecular re-arrangement in aromatic amines,

## CORRESPONDENCE.

ESTIMATION OF THE ADULTERANT IN  
CITRONELLA OIL.*To the Editor of the Chemical News.*

SIR,—In the CHEMICAL NEWS of January 8, 1904 (vol. lxxxix., p. 21), there is an article by M. K. Bamber on the "Estimation of the Adulterant in Citronella Oil." I would like to know where the tubes there described may be obtained, and if the specific gravity of the alcohol, 0.8273 at 30° C., is referred to water at 15° C. in air as unity.—I am, &c.,

WILSON H. LOW.

Chemist of the Cudahy Packing Co.

The Cudahy Packing Co., South Omaha, Neb.  
January 22, 1904.

## THEORY OF ELECTROLYTIC DISSOCIATION.

*To the Editor of the Chemical News.*

SIR,—As already stated a few weeks back, I propose shortly to contribute, with your permission, a few pages on the nature of the liquid state, and the position I shall therein take up is one diametrically opposed to the theory advocated in Mr. Joseph W. Richards's recent paper (CHEMICAL NEWS, vol. lxxxix., pp. 31, 37).

But meanwhile, lest your readers should be over-impressed by the calculations which form the sole appeal made by Mr. Richards to experimental facts, permit me to offer a few remarks in criticism of the same.

It is certainly a very remarkable coincidence that the concentration used by Thomsen in the determination of the heat of neutralisation of caustic soda by hydrochloric acid should be just the one which favours Mr. Richards's theory. Yet so it is, and a little consideration will show that with any other concentration his figures totally fail.

To prove this, I must quote his calculations pretty fully. He contends that, according to his theory, the heat of neutralisation must be equal to the heat of vaporisation of liquid water at the given temperature, so as to form gaseous water at the same temperature and under an osmotic pressure equal to that due to the "ionised water," from which he assumes it is formed. The temperature is 18°, and the osmotic pressure of "ionised water" under the conditions of concentration assumed by Thomsen would be, as Mr. Richards shows, about 6.19 atmospheres. He uses this pressure to calculate the total work of vaporisation, taking the heat of vaporisation less the "outer work" as 10,110 calories (obtained quite legitimately from Regnault's figures), and adding thereto the outer work  $6.19 \times$  the thermal factor  $2 \times T$ . Thus he obtains the number  $10,110 + 6.19 \times 2T = 10,110 +$  (not  $\times$  as misprinted)  $3590 = 13,700$  calories. Now the heat of neutralisation is 13,740 calories. Q.E.D.

Yes, but let us assume another concentration. Let us take instead of Thomsen's concentration a solution ten times as dilute. Now, everybody knows well enough that Thomsen used a dilution so great that further dilution made practically no thermal difference. At this greater dilution, then, the heat of neutralisation will still be 13,740. But the osmotic pressure above referred to must be reduced to  $\frac{1}{10}$ ; it is now only 0.619 atmosphere, and according to Mr. Richards's calculation the result will come out  $10,110 + 0.619 \times 2T = 10,110 + 3.59 = 10,114$  calories. So much for Q.E.D.

And then again in his next calculation he uses this same quantity (less one atmosphere in his product), viz.,  $(6.19 - 1) \times 2T = 3021$  calories, to add to his calculated number 55,200 in order to make the result come out to 58,221 calories, and thus tally with the molecular heat of formation of water gas from oxygen and hydrogen which, as deduced from Regnault, is 58,100 calories.

But under the circumstances of greater dilution as above, the osmotic pressure is not 6.19 atmospheres but 0.619, and the quantity to be added will be negative, viz.,  $(0.619 - 1) \times 2T = -0.381 \times 2T = -222$ . The result instead of 58,100 is 54,978. And with other concentrations of course other variations occur. In fact, by diminishing the concentration and increasing the temperature, you can gradually make the number 55,200 to vanish. Thus does the sole experimental proof of the theory for which such an imposing array of tabulated figures are deduced collapse like a house of cards.

Mr. Richards's theory is originated, he tells us, to account for the fact that that one molecule of a salt (say KCl) cannot be ionised (into two molecules K, Cl) without enormous thermal disturbance. But if this is any difficulty it is entirely balanced by the fact that the electrolytic decomposition of the ionised KCl into free potassium and free chlorine takes place practically without any expenditure of work. That is a fact really quite inexplicable on Mr. Richards's theory, so he only surmounts one difficulty to raise another. If the point he refers to is really difficult, the difficulty readily vanishes on consideration of the enormous tensions (not osmotic but due to intermolecular attraction), hundreds of atmospheres, which exist between the molecules of the solvent, and the mechanical changes in these tensions, resulting from the introduction of the solute, are quite sufficient to account for the disappearance of the heat of decomposition of the salt into its ions.

There is another point, however. If the ionised salts are, as Mr. Richards says, still compounds, then since, as he points out, they have the properties of a gas (even the osmotic coefficient agreeing with the gas coefficient), how comes it that an ionised binary molecule, such as KCl, poses as two gas molecules in its osmotic pressure and not as one?—I am, &c.,

P. J. BEVERIDGE, M.A., B.Sc.

7, Bridge Place, Chester,  
February 1, 1904.CHEMICAL NOTICES FROM FOREIGN  
SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 2, January 11, 1904.

**Colour Reactions of Vanadic Acid and Ethanol.**—Camille Matignon.—Whilst investigating a method of colour estimation of vanadic acid the author discovered a number of facts about the properties of this acid. Tannin gives a dark blue precipitate or a colouration of the same tint with solutions of ammonia metavanadate when fairly dilute, and the author finds that gallic and pyrogallous acid with vanadic solutions also give very dark blue colourations. He also makes various experiments on certain active ethers and their effects on vanadic acid.

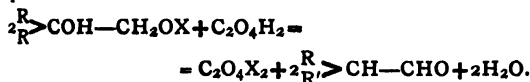
**Use of Bismuth as a Separating Agent in the Series of the Rare Earths.**—G. Urbain and H. Lacombe.—Already inserted in full.

**New Method of Estimation of the Halogen Elements in Organic Bodies.** Chlorine and Bromine.—H. Baubigny and G. Chavanne.—In a previous research the authors investigated a method of estimation of iodine in organic compounds, based on the destruction of the substance by a chromosulphuric mixture and the transformation of the iodine into fixed iodic acid. It is evident that the same method of combustion can be applied to bodies containing chlorine and bromine, but the method fails when the oxidation of the chlorine or bromine is attempted. However, they describe, with a drawing, an apparatus by which the quantitative estimation of the chlorine and bro-

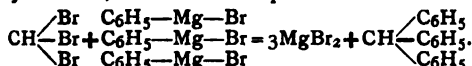
mine, after the destruction of the organic matter, can be accurately determined.

**Titration of Manganese.**—Léon Débourdeaux.—The titration of manganese oxides necessitates two determinations—(1) That of the chlorine which the oxide can set free; (2) that of the hydrochloric acid necessary to set free all the chlorine which is present. These two estimations, which usually need two distinct operations, can be effected in a single experiment by a method founded on the destruction of the superior oxides of manganese by hot oxalic acid in presence of suitably diluted sulphuric acid. This method has the advantage of needing very little attention. The two determinations of the titration take place rapidly and exactly in a single experiment. It is applicable to all manganese salts without previous elimination of the carbonates.

**A Method of Synthesis of Aldehydes.**—MM. Béhal and Sommelet.—The authors find that the aldehydes  $(R)_2=CH-CHO$  and  $R_1R_2C=CH-CHO$  can be obtained from the ether oxides of the  $\alpha$ -glycols corresponding to the formulæ  $\frac{R}{R}COH-CH_2OX$  and  $\frac{R}{R}COH-CH_2OX$  by decomposition with dry oxalic acid. In these formulæ, X represents an alcoyl or a monovalent aromatic radical. The transformation is effected according to the following equation:—



**Synthesis of Aromatic Aldehydes.**—F. Bodroux.—Iodoform and bromoform act energetically on organo-magnesium compounds at ordinary temperatures. In the case of phenylmagnesium bromide the author obtained triphenylmethane, as well as other products:—



The yield in this case was not good, so he replaces the halogen derivatives by ethyl-ortho-formiate, and, at the temperature of a water-bath after six hours, a considerable quantity of benzylic aldehyde is produced.

**Active Influence of Albumenoid Matter on Oxidation by means of Manganese.**—A. Trillat.—The author's experiments show that albumin has the property of preventing the precipitation of manganese dioxide even in presence of a strong alkali. If a solution of a manganese salt and an alkali are successively added to a dilute solution of albumin the liquid becomes slightly brown, but no precipitate forms; if, on the contrary, there is no albumin, an abundant precipitation takes place.

## MISCELLANEOUS.

**Some Constants of Sulphide of Carbon.**—V. Unruh.—The sulphide is purified by treating 500 to 700 c.c. with 6 to 8 c.c. of pure dry mercury and porous  $\text{CaCl}_2$ . Shake, filter, and distil in the dark. This operation is repeated until no more sulphide of mercury is formed. The first portions (15 to 20 per cent) and the last portions (10 to 15 per cent) are set on one side, and treated again like the impure sulphide. The pure product has an ethereal odour, and its boiling-point is constant to about  $0.002^\circ$ ; this is contrary to the results found by Arctowsky (*Zeit. Anorg. Ch.*, vol. vi., p. 255). A variation of 1 m.m. in the atmospheric pressure causes a variation of  $0.04144^\circ$  in the boiling-points. At a boiling-point of  $46.25^\circ$ , the density = 1.2209. This density varies considerably when the boiling takes place under pressures slightly different from the atmospheric pressure. For example, at 756.6 m.m., and with a boiling-point of  $46.10^\circ$ , the density = 1.22161.—*Zeit. Anorg. Ch.*, vol. xxxii., p. 407.

**Institute of Chemistry Examinations.—Pass Lists** (January, 1904).—*Intermediate Examination*: Stanley Winter Collins, King's Coll., London; David Cowan Crichton, Univ. Coll., Dundee; Harold Deane, B.Sc. (Lond.), School of the Pharmaceutical Society, London; Cyril Dickinson B.Sc. (Vict.), Yorkshire Coll., Leeds; John Augustus Goodson, Finsbury Tech. Coll., London; Henry Ward Goodwin, Univ. Coll., Nottingham; Alfred George Holborow, Merchant Venturers' Tech. Coll., Bristol, and Glasgow and W. of Scot. Tech. Coll.; Charles James, Univ. Coll., London; Harry Edwin Laws, Univ. Coll., Nottingham; Robert Park, King's Coll. London, and under J. Falconer King, F.I.C.; Edwin Rhodes, B.Sc. (Vict.), Yorkshire Coll., Leeds. *Final A.I.C. Examination—Branch "A" (Mineral Chemistry)*: Denison Benzeville Byles, Univ. Coll., London; Arthur Charles Carter, Univ. Coll., London; James Gray, Glasgow and W. of Scot. Tech. Coll.; Arthur Hopwood, Assoc.R.C.Sc. (Lond.), Royal Coll. of Science, London. *Branch "D" (Organic Chemistry)*: Marmaduke Barrowcliff, Univ. Coll., Nottingham; Edwin Jesse Fairhall, A.C.G.I., Central Tech. Coll., London; William Oswald Littlebury, Univ. Coll., Nottingham; Miss Frances Mary Gore Micklethwait, Assoc.R.C.Sc. (Lond.), Royal Coll. of Science, London; Fred. Scholefield, B.Sc. (Lond. and Vict.), Yorkshire Coll., Leeds; Harold Stevenson, Univ. Coll., Nottingham. *Branch "E" (Analysis of Food and Drugs and of Water, including Examination in Therapeutics, Pharmacology, and Microscopy)*: James Handby Ball, B.Sc. (Vict.), Univ. Coll., Liverpool; Herbert Sutcliffe Shrewsbury, Univ. Coll., Nottingham; Henry Edgar Watt, M.Sc. (Durham), Durham Coll. of Science, Newcastle-on-Tyne. *For the Fellowship (F.I.C.)—Branch "D" (Organic Chemistry)*: Harold Hibbert, M.Sc. (Vict.), Owens Coll., Manchester, and Univ. Coll., Aberystwyth. *Branch "E" (Analysis of Food and Drugs and of Water, including Examination in Therapeutics, Pharmacology, and Microscopy)*: Edward William Lucas, School of the Pharmaceutical Society, London.

**Organo-metallic Compounds of Benzoic Acid.**—L. Pesci.—*o*-Oxymercuribenzoic Anhydride,  $\text{C}_6\text{H}_4\langle\frac{\text{CO}}{\text{Hg}}\rangle\text{O}$ .—This substance is obtained by heating a mixture of 32 parts of acetate of mercury and 20 parts of benzoic acid to about  $140^\circ$ . The mass is dissolved in a boiling solution of  $\text{Na}_2\text{CO}_3$ , and the solution precipitated with  $\text{CO}_2$ ; the precipitate dissolved in ammonia gives the ammonium salt in a well crystallised form; this is decomposed by dilute acetic acid. It is also obtained by boiling acetate of mercury with phthalate of soda and a little acetic acid.  $\text{CO}_2$  is given off, and the mercury is substituted for the ortho,  $\text{CO}_2\text{H}$ . It is an insoluble white powder, decomposed by the mineral acids. When strongly heated it deflagrates, *Salt of Ammonium*,  $\text{HO}-\text{C}_6\text{H}_4-\text{CO}_2\text{NH}_4$ .—Needles, slightly soluble in water in the cold, dissociated by heat, returning to the anhydride. The salt of barium is obtained from  $\text{Ba}(\text{OH})_2$  and the anhydride. It occurs in needles, soluble in water, decomposed by  $\text{CO}_2$ . The salts of calcium, magnesium, isoamylamine, and benzylamine are also formed. *o*-Chloromercuribenzoic Acid,  $\text{C}_6\text{H}_4\langle\frac{\text{CO}_2\text{H}}{\text{HgCl}}\rangle$ .—This is a microcrystalline powder, slightly soluble in water, obtained by precipitating one of the preceding salts with acetic acid in the presence of a large excess of a soluble chloride. The salt of potassium,  $\text{C}_6\text{H}_4(\text{HgCl})(\text{CO}_2\text{K})\cdot\frac{1}{2}\text{H}_2\text{O}$ , is obtained in brilliant needles by the saturation of a hot solution of KCl with mercurio-benzenic anhydride; it is deposited on cooling; it loses its water at  $100^\circ$ , and is decomposed by hot water. There are also formed the salt of soda with  $2\frac{1}{2}\text{H}_2\text{O}$ ; the salt of ammonium crystallised with  $2\text{NH}_4\text{Cl}$ ; the salt of barium with  $3\text{H}_2\text{O}$ , and the salt of aniline. *o*-Bromomercuribenzoic Acid,  $\text{C}_6\text{H}_4(\text{CO}_2\text{H})(\text{HgBr})$ .—The salt of potassium, the salt of sodium with  $3\text{H}_2\text{O}$ , and the salt of barium with  $3\text{H}_2\text{O}$  are all prepared as in the preceding cases. *o*-Iodo-

**mercuriobenzoic Acid.**—This forms anhydrous salts of potassium, sodium, and barium. *The Salts of o-Sulpho-mercuriobenzoic Acid*,  $\text{S}:(\text{Hg}.\text{C}_6\text{H}_4.\text{CO}_2\text{H})_2$ .—The salt of sodium is obtained by the action of 1 molecule of  $\text{Na}_2\text{S}$  on 2 molecules of mercuribenzoate of soda. The precipitate consists of white needles. Acetic acid gives a gelatinous mass; the salt of potassium is in needles, soluble in water. *o-Mercuriodibenzoic acid*,  $\text{Hg}:(\text{C}_6\text{H}_4.\text{CO}_2\text{H})_2$ , obtained by the prolonged boiling of one of the preceding sulpho-salts with water,  $\text{HgS}$  is produced, and the corresponding salt of the acid. Acetic acid decomposes this salt and precipitates the free acid, which is crystallised in boiling alcohol. It occurs in colourless needles decomposed by heat. The salt of potassium is in needles soluble in water; that of sodium is slightly crystalline; that of ammonium occurs in rhombohedra slightly soluble in water, and that of calcium is insoluble in water.—*Gazz. Chim. Ital.*, [2], vol. xxxii., p. 277.

### MEETINGS FOR THE WEEK.

**MONDAY, 15th**—Society of Arts, 8. (Cantor Lectures). "Oils and Fats—their Uses and Applications," by J. Lowkowitzsch, Ph.D., &c.

**TUESDAY, 16th.**—Royal Institution, 5. "Development of Animals," by Prof. L. C. Miall, F.R.S.

**WEDNESDAY, 17th**—Society of Arts, 8. "Garden Cities in their Relation to Industries and Agriculture," by A. R. Sennett.

Chemical, 5.30. "Observations on some Continuous Intramolecular and at first Reversible Changes extending over Prolonged Periods of Time," by R. J. Friessell. "The Esterification of  $\alpha$ -Mandelic Acid by Menthyl and Borneol," by A. McKenzie.

Microscopical, 8. "On the Vertical Illuminator" and "The Influence of the Antipoint on the Microscopic Image shown Graphically," by E. M. Nelson. "A Microscope with Geometric Slides," by Keith Lucas. Mr. C. L. Curtis will exhibit Specimens of Marine Objects mounted by Mr. H. J. Waddington.

**THURSDAY, 18th.**—Royal Institution, 5. "Recent Research in Agriculture," by A. D. Hall, M.A.

**FRIDAY, 19th.**—Royal Institution, 9. "Condensation Nuclei" (Illustrated by Experiment), by C. T. R. Wilson, F.R.S.

**SATURDAY, 20th.**—Royal Institution, 3. "The Life and Work of Stokes," by Right Hon. Lord Rayleigh, F.R.S.

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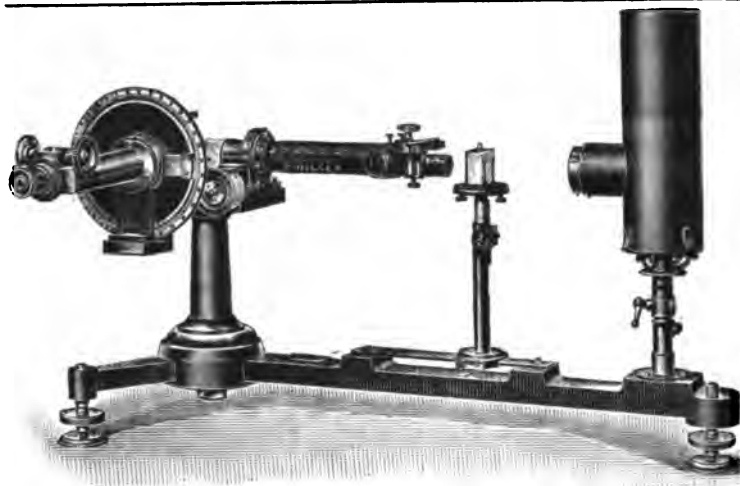
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## CONTENTS.

| ARTICLES:—                                                                                                                                                                                                                     | PAGE |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| Examination of a Sample of Gas occluded in Radium Bromide, by Professors Curie and Dewar .....                                                                                                                                 | 85   |
| On the Compressibilities of Oxygen, Hydrogen, Nitrogen, and Carbonic Oxide between One Atmosphere and Half an Atmosphere of Pressure, and on the Atomic Weights of the Elements concerned, by Lord Rayleigh, O.M., F.R.S. .... | 86   |
| On certain Properties of the Silver-cadmium Series of Alloys, by T. Kirke Rose, D.Sc. ....                                                                                                                                     | 86   |
| Production of Crystallised Salts of Bismuth, by A. de Schuiten..                                                                                                                                                               | 87   |
| London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S. ....                                                                                                                                              | 88   |
| Precipitation and Separation by Weak Organic Bases, by E. T. Allen .....                                                                                                                                                       | 88   |
| On the Colorimetric Determination of Small Quantities of Phosphoric Acid and of Silica, by F. T. Veitch .....                                                                                                                  | 89   |
| What Physical Chemistry has done for Chemistry, by H. C. Jones                                                                                                                                                                 | 91   |
| PROCEEDINGS OF SOCIETIES—                                                                                                                                                                                                      |      |
| CHEMICAL SOCIETY .....                                                                                                                                                                                                         | 92   |
| PHYSICAL SOCIETY .....                                                                                                                                                                                                         | 93   |
| NOTICES OF BOOKS .....                                                                                                                                                                                                         | 95   |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                                                                                                    | 95   |
| MISCELLANEOUS .....                                                                                                                                                                                                            | 95   |
| MEETINGS FOR THE WEEK .....                                                                                                                                                                                                    | 96   |

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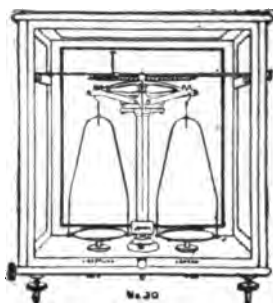
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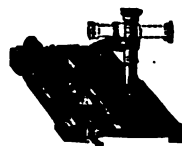


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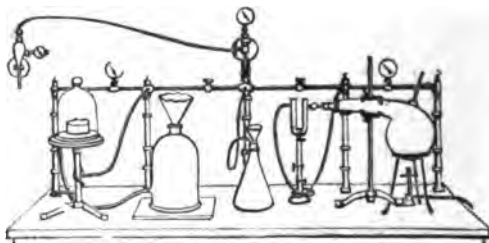
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VOL. LXXXIX., No. 2308.

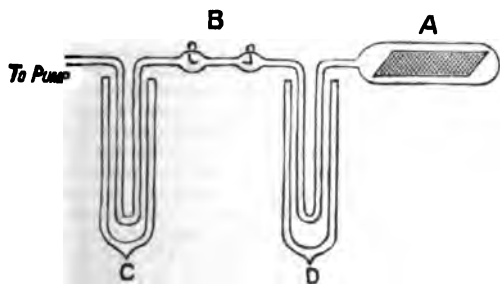
## EXAMINATION OF A SAMPLE OF GAS OCCLUDED IN RADIUM BROMIDE.\*

By Professors CURIE and DEWAR.

THE sample of radium bromide amounting to 0.42 grm. had been prepared about three months, and was brought to the Royal Institution for the purpose of continuing the enquiry on the heat evolution at the temperature of boiling hydrogen. It contained some water of hydration, and had been giving off permanent gas amounting to about 1 c.c. per month while kept in a chamber that had been previously highly exhausted. This gas, examined in the spectroscope, appeared to be chiefly hydrogen.

It was now determined to transfer the specimen of the radium bromide to a quartz tube, so that it might safely be exhausted to the limit of a mercurial vacuum during the fusion of the salt above a red heat. By introducing two or three small U-tubes immersed in liquid air between the pump and the quartz tube containing the salt, all mercurial vapour was prevented from access to the quartz tube, while water, together with traces of bromine or carbonic acid, and the emanation given off during the heating, were separated and condensed. At the same time any permanent gas passing the liquid air condensers, along with the more volatile part of the emanation pumped out during the continuance of the high exhaustion, was collected after passing through the mercurial pump.

Having exhausted the radium bromide for some hours at the ordinary temperature until no further trace of gas came off, it was gradually heated to fusion. During the heating, gas came off rapidly, the total quantity collected amounting to 2.6 c.c. (in all probability not less than thirty times the volume of the fused radium bromide). After the elimination of gas from the fused radium bromide had ended, the small piece of quartz tube, containing now about 0.4 grm., was sealed off by means of the oxy-hydrogen blow-pipe, the intention being to examine it later on spectroscopically.



The gas that came through the mercurial pump was transferred to a small mercury burette, and as it was so luminous and powerfully radio-active it was put away in a dark room for the purpose of taking a photograph of its spectrum. On developing the plate after three days' exposure (during which time the walls of the glass tube containing it had become deep violet and nearly half the volume of gas absorbed) it was found to reveal a discontinuous spectrum, consisting of three bands coincident with the nitrogen bands  $\lambda$  3800, 3580, and 3370. This is a remarkable result considering

the amount of the emanation present could only be small, being such as was volatile at the temperature of liquid air.

The part of the eudiometer where the luminous gas had stood over mercury was cut off. The glass was deep violet all through the mass, and the surface was covered in parts with what looked like some mercury compound. This piece of tube was placed inside a closed tube, A, of hard glass, which was sealed on to a vacuum tube, B, with small U-tubes, C and D, on each side for cooling in liquid air.

After thorough exhaustion the part A was heated to a low red heat, so that the violet coloured piece of tube covered with the mercury compound might be decomposed and any permanent gaseous product collected in a glass vacuum tube, while vapour of water, carbonic acid, and mercury vapour were all condensed in the liquid air U-tubes. The result of the heating was to make the glass lose its violet colour and to increase the pressure in the sparking tube from the gas given off during the heating. The vacuum tube was afterwards sealed off from the U-tubes and spectroscopically examined. This gas gave the spectrum of nitrogen, no hydrogen being visible.

To confirm the composition of the gas, occluded by the radium bromide, that portion which had not been absorbed while standing over mercury was transferred to a highly exhausted spectroscopic tube having a narrow side tube attached that could be cooled in liquid hydrogen and then sealed off. In the transference of the gas and exhaustion of the vacuum tube, liquid air U-tubes to condense impurities and keep out mercurial vapour were employed.

On passing the electric discharge, the spectrum emitted was solely that of the nitrogen band spectrum, no other lines being visible. When the side tube was placed in liquid hydrogen the nitrogen rapidly condensed, and the vacuum became very high. After the condensed material in the side tube was sealed off, the vacuum tube showed a hydrogen spectrum, and nothing else. In such a tube it would be impossible to find small amounts of helium by the spectroscope, seeing that a 10 per cent helium-hydrogen mixture shows nothing but hydrogen.

The most hopeful anticipation of a knowledge of the character of the gases given off during the transitions taking place in radium bromide was clearly to make a spectroscopic examination, after keeping for some time, of any gaseous atmosphere developed in the carefully exhausted quartz tube, and this M. Deslandres has done, with the result that the internal gas, illuminated by an induction spark through two pieces of tin-foil covering the two ends of the tube, gave the entire spectrum of helium. No lines except those of this gas were discovered after an exposure of three hours in a quartz photographic spectroscope.

Until the full spectroscopic examination by Deslandres is published no inference can be drawn as to whether or no the amount of helium has gone on increasing in the quartz tube (as it ought to do if it is a true product of the disintegration of radium) and no conclusion drawn as to the presence of other gases and their origin, whether new or old.

During the discussion of radio-activity at the last meeting of the British Association, an account was given of some experiments made by Prof. Dewar and Sir William Crookes on the spectrum given by radium bromide when sealed up in quartz tubes, in the first instance when exhausted, and, secondly, when filled with helium at different pressures. In neither case did a discontinuous spectrum result. If the gas in presence of the radium bromide was, however, air, the nitrogen bands appeared under exactly the same conditions, thus confirming the observations of Sir W. and Lady Huggins. It is an interesting fact that the radiations should not be able to excite the helium atom to vibration, whilst this result is easily attained in the case of nitrogen. Further, helium at atmospheric pressure seems to be the gas of all others that most easily conducts electric discharges. This matter requires further investigation.

\* Translated from the *Comptes Rendus*, vol. cxxxviii., p. 190, with emendations.

ON THE COMPRESSIBILITIES OF OXYGEN,  
HYDROGEN, NITROGEN, AND CARBONIC OXIDE  
BETWEEN ONE ATMOSPHERE AND  
HALF AN ATMOSPHERE OF PRESSURE,  
AND ON THE ATOMIC WEIGHTS OF THE  
ELEMENTS CONCERNED.\*

(PRELIMINARY NOTICE).

By Lord RAYLEIGH, O.M., F.R.S.

THE observations now referred to were conducted with an apparatus designed upon the same lines as that already described ("On the Law of the Pressure of Gases between 75 and 150 m.m. of Mercury," *Phil. Trans.*, A., 1902, vol. 198, pp. 417-430). It must suffice to mention that the only important modification lay in the fact that the two single volumes, which, when employed together, constitute the double volume, were used separately and alternately, so as to eliminate in each set of measurements any question as to what the ratio of these volumes exactly is. It is hoped to give a full description of the method when it has been extended to the examination of other gases, such as nitrous oxide and carbonic anhydride. The temperatures ranged from 10°-15°, and care was taken that in each measurement the mean temperatures should be almost exactly the same for the single and for the double volume.

The results were reduced much as previously explained, and give for the values of  $B$ , which, according to Boyle's law, should be unity:—

|                     |         |
|---------------------|---------|
| Oxygen .. .. .      | 1.00040 |
| Hydrogen .. .. .    | 0.99976 |
| Nitrogen .. .. .    | 1.00017 |
| Carbonic oxide .. . | 1.00028 |

$B$  here denotes the quotient of the value of  $pV$  at the half atmosphere by the corresponding value at the whole atmosphere. That it would be less than unity in the case of hydrogen, and exceed unity for the other gases, is what would be anticipated from their behaviour at higher pressures.

If we measure  $p$  in atmospheres, and assume, as has usually been done, e.g., by Regnault and Van der Waals, that at small pressures the equation of an isothermal is:—

$$pV = PV(1 + ap),$$

where  $PV$  is the value of the product in a state of infinite rarefaction, then:—

$$a = 2(1 - B).$$

Probably the chief interest of a knowledge of the coefficient  $a$  is the application to deduce a correction to the relative densities of gases as observed at atmospheric pressure, so as to determine what would be the relative densities in a state of great rarefaction, to which alone Avogadro's law is applicable. (The application to oxygen and hydrogen was made in my paper, "On the Relative Densities of Oxygen and Hydrogen," *Roy. Soc. Proc.*, 1892, vol. I., p. 448; "Scientific Papers," vol. iii., p. 525).

Taking oxygen as a standard, we see that the small correcting factor to be introduced in order to pass from the ratio of densities at one atmosphere to that at great rarefaction, is  $(1+a)/(1+a_0)$ , or  $1+2(B_0-B)$ , the suffix 0 relating to oxygen, that is, as follows:—

|                     |         |
|---------------------|---------|
| Hydrogen .. .. .    | 1.00128 |
| Nitrogen .. .. .    | 1.00046 |
| Carbonic oxide .. . | 1.00024 |

The double of the first number, viz., 2.0026, represents, according to Avogadro's law, the volume of hydrogen which combines with one volume of oxygen at atmospheric pressure to form water. Direct determinations by Scott gave 2.00245, and Morley, in his later work, found 2.0027, so that there is here a good agreement.

The following table gives the densities of the various gases, referred to oxygen = 16, at atmospheric pressure and at very small pressure, as deduced from my own observations (*Roy. Soc. Proc.*, 1893, vol. liii., p. 134; 1897, vol. lxi., p. 204; "Scientific Papers," vol. iv., pp. 39, 352):—

| Gas.                | Atmospheric pressure. | Very small pressure. |
|---------------------|-----------------------|----------------------|
| Hydrogen .. .. .    | 1.0075                | 1.0088               |
| Nitrogen .. .. .    | 1.0003                | 1.0009               |
| Carbonic oxide .. . | 1.0000                | 1.0003               |

From the researches of M. Leduc and Prof. Morley, it is probable that the above numbers for hydrogen are a little, perhaps one thousandth part, too high.

The uncorrected number (1.0003) for nitrogen has already been given (Rayleigh and Ramsay, *Phil. Trans.*, A., 1895, vol. clxxxvi., p. 187), and contrasted with the 1.005 obtained by Stas. This question deserves the attention of chemists. If Avogadro's law be strictly true, it seems impossible that the atomic weight of nitrogen can be 14.05.

From the molecular weight of CO, viz., 28.006, we deduce, as the atomic weight of carbon, 12.006.

It should be mentioned that D. Berthelot (*Comptes Rendus*, 1898) has, meanwhile, calculated very similar numbers, based upon the observations of Leduc.

ON CERTAIN PROPERTIES OF THE  
SILVER-CADMIUM SERIES OF ALLOYS.\*

By T. KIRKE ROSE, D.Sc.

THE attempts made at the Royal Mint to produce uniform standard trial plates of silver and copper have been unsuccessful owing to the segregation of the constituents. The cooling curve of the alloy shows that solidification begins at 900° and ends at 778°, after passing through a pasty stage, during which rearrangement of the constituents can take place, with the result that the uniform distribution of the silver is disturbed. The cooling curve of the alloy containing 92.5 per cent of silver and 7.5 per cent of cadmium is found to resemble that of a pure metal, showing no appreciable pasty stage, and on testing plates made of these materials, they were found to be uniform in composition. The alloy is exceedingly ductile, and no difficulty is encountered in making assays on it by any of the well-known methods. In preparing large ingots it is necessary to pour silver into a suitable amount of molten cadmium, this method minimising the loss of cadmium by volatilisation.

The cooling curves and the microstructure of the whole series of alloys of silver and cadmium have also been studied, and evidence has been obtained of the existence of a number of compounds. The curves of equilibrium of the series are complicated by the effects of the compounds. The alloys containing from 100 to 80 per cent of silver are homogeneous at all temperatures below the solidus curve, although they appear to contain two bodies between the solidus and liquidus curves. There is a well-marked cusp in the curves, corresponding to the compound  $Ag_2Cd_3$  and an alloy of minimum freezing point containing about 1 per cent of silver. Among the alloys containing less than 80 per cent of silver, only those corresponding in composition to the formulæ  $Ag_2Cd$ ,  $AgCd$ ,  $Ag_2Cd_3$ , and  $AgCd_3$  appear to be homogeneous.

Synthesis of Sugars from Trioxymethylene and Sodium Sulphite.—A. Seyewetz and M. Gibello.—The authors transform trioxymethylene dissolved in a solution of sodium sulphite into sugar derivatives. No appreciable transformation takes place at ordinary temperatures, but sugar compounds begin to be formed directly the solution is heated on the water-bath, and more rapidly still when boiled.—*Comptes Rendus*, cxxxviii., No. 3.

\* A Paper read before the Royal Society, February 11, 1904.

\* Abstract of a Paper read before the Royal Society, Feb. 1, 1904.

# THE PRODUCTION OF CRYSTALLISED SALTS OF BISMUTH.

By A. DE SCHULTEN.

## Sub-nitrates of Bismuth.

In undertaking the examination of these salts, which have been already the subject of research by numerous writers, I have limited the field of my inquiry at first to the crystalline compounds which are produced by the action of water in the cold on a solution of nitrate of bismuth in nitric acid.

**The Salt  $5\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ .**—I dissolved 50 grms. of the salt  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  in 50 c.c. of nitric acid of 1·2 density, and added 3 litres of water while stirring. After twelve hours I collected the crystals produced by this reaction on a filter, and pressed them between filter-papers with great care. The crystals are air-dried until their weight no longer changes. During this desiccation the crystals do not undergo any change. They retain their form and limpidity, and their facets, though striated, always remain smooth.

On analysis the crystals give the following figures:—

|                                 | Found. |       |       | Calculated for<br>$5\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ . |
|---------------------------------|--------|-------|-------|----------------------------------------------------------------------------------------------------|
|                                 | I.     | II.   | III.  |                                                                                                    |
| $\text{Bi}_2\text{O}_3$ .. ..   | 76·83  | 76·83 | 76·84 | 76·79                                                                                              |
| $\text{N}_2\text{O}_5$ .. ..    | 17·82  | —     | —     | 17·85                                                                                              |
| $\text{H}_2\text{O}$ (by diff.) | 5·35   | —     | —     | 5·36                                                                                               |
|                                 | 100·00 |       |       | 100·00                                                                                             |

| Calculated for—                                                                  |                                                                                   |                                                                                |        |
|----------------------------------------------------------------------------------|-----------------------------------------------------------------------------------|--------------------------------------------------------------------------------|--------|
| $4\text{Bi}_2\text{O}_3 \cdot 4\text{N}_2\text{O}_5 \cdot 7\text{H}_2\text{O}$ . | $6\text{Bi}_2\text{O}_3 \cdot 6\text{N}_2\text{O}_5 \cdot 11\text{H}_2\text{O}$ . | $\text{Bi}_2\text{O}_3 \cdot \text{N}_2\text{O}_5 \cdot 2\text{H}_2\text{O}$ . |        |
| $\text{Bi}_2\text{O}_3$ .. ..                                                    | 76·91                                                                             | 76·72                                                                          | 76·34  |
| $\text{N}_2\text{O}_5$ .. ..                                                     | 17·88                                                                             | 17·83                                                                          | 17·74  |
| $\text{H}_2\text{O}$ .. ..                                                       | 5·21                                                                              | 5·45                                                                           | 5·92   |
|                                                                                  | 100·00                                                                            | 100·00                                                                         | 100·00 |

The oxide of bismuth was estimated by calcining the material, and the nitric acid by Pelouze's method. In Analysis I. the oxide of bismuth was estimated on 12·87 grms. of material; in Analysis II. on 8·04 grms. of material from another preparation. In Analysis III. the bismuth was estimated on 3·78 grms. of material. For this analysis I prepared the substance in the manner just described, with the exception that I precipitated the solution of nitrate of bismuth with only 1·75 litres of water instead of 3 litres, and I collected the crystals immediately after their formation. In this manner I proved conclusively that the contact of the crystals with the mother-liquor for twelve hours did not cause any partial decomposition.

The figures furnished by the analyses do not agree, as will be easily seen, with those calculated for the simple formula  $\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ , generally accepted for the sub-nitrate of bismuth obtained under the conditions in which I operated. The formula which represents their composition the best is  $5\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ .

When placed over sulphuric acid the crystals lose water. After forty days their weight remained constant. The loss of weight amounted to 3·14 per cent, corresponding to 5·27 molecules of water.

The crystals have a pearly lustre. They take the form of very thin plates, hexagonal in form, and more often slightly elongated. They are striated parallel with the elongation. Extinction takes place at an angle of about 13° with the elongation. The density of the crystals, determined on 5·45 grms. of material, was found to be 4·928 at 15°. They dissolve in water, and the solution deposits crystals of the following salt.

**The Salt  $5\text{Bi}_2\text{O}_3 \cdot 4\text{N}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$ .**—If we add water to the mother-liquors from the preceding crystals, we obtain this salt in a well-crystallised form. We do not obtain any other compound than this, no matter what quantity of

water may be added. By mixing 5 litres of water with 2 litres of the mother-liquor of the preceding crystals, I obtained, after eight days, measurable crystals of this salt.

These crystals are not attacked by water, and they do not undergo any change in free air or over sulphuric acid.

The analysis of these crystals, dried in the air until their weight is constant and carried out in the same manner as in the preceding case, gave the following figures:—

|                                 | Found. | Calculated for—                                                                  |                                                                                  |
|---------------------------------|--------|----------------------------------------------------------------------------------|----------------------------------------------------------------------------------|
|                                 |        | $5\text{Bi}_2\text{O}_3 \cdot 4\text{N}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$ . | $5\text{Bi}_2\text{O}_3 \cdot 4\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ . |
| $\text{Bi}_2\text{O}_3$ .. ..   | 80·05  | 80·13                                                                            | 79·64                                                                            |
| $\text{N}_2\text{O}_5$ .. ..    | 14·87  | 14·90                                                                            | 14·81                                                                            |
| $\text{H}_2\text{O}$ (by diff.) | 5·08   | 4·97                                                                             | 5·55                                                                             |
|                                 | 100·00 | 100·00                                                                           | 100·00                                                                           |

|                               |  | Calculated for—                                                                  |                                                                                  |
|-------------------------------|--|----------------------------------------------------------------------------------|----------------------------------------------------------------------------------|
|                               |  | $6\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$ . | $6\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ . |
| $\text{Bi}_2\text{O}_3$ .. .. |  | 80·30                                                                            | 79·89                                                                            |
| $\text{N}_2\text{O}_5$ .. ..  |  | 15·55                                                                            | 15·47                                                                            |
| $\text{H}_2\text{O}$ .. ..    |  | 4·15                                                                             | 4·64                                                                             |
|                               |  | 100·00                                                                           | 100·00                                                                           |

The oxide of bismuth was estimated by the calcination of 6·23 grms. of material air-dried until its weight no longer varied. When dried over sulphuric acid the salt experienced a loss of 0·12 per cent. My colleague, F. Pisani, was kind enough, at my request, to make a control estimation of the nitric acid by Pelouze's method on a sample from another preparation. He found  $\text{N}_2\text{O}_5 = 14·76$  per cent.

The composition of the crystals thus approaches closest to the formula  $5\text{Bi}_2\text{O}_3 \cdot 4\text{N}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$ . For bodies obtained under similar conditions to those which caused the formation of these crystals, the provisional formulæ  $5\text{Bi}_2\text{O}_3 \cdot 4\text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$  or  $6\text{Bi}_2\text{O}_3 \cdot 5\text{N}_2\text{O}_5 \cdot 8\text{H}_2\text{O}$  (or  $9\text{H}_2\text{O}$ ) have been proposed.

The crystals are limpid and brilliant, and take the form of rectangular tables up to 1·6 m.m. in length, 0·3 m.m. in width, and 0·03 m.m. in thickness. They are monoclinic. The faces,  $p$  (001) dominant,  $o^1$  (101) and  $e^1$  (011) are observed. Only two faces  $e^1$  (011), are seen. I have measured the following normal angles with the goniometer:  $p$   $o^1$  (001) (101) = 52° 2' and  $p$   $e^1$  (001) (011) = 59° 30'.

On  $p$  (001) and  $o^1$  (101) the extinctions are longitudinal. With a strip cut perpendicularly to  $p$  (001), extinction takes place at an angle of about 40° with the edge  $p$   $g^1$  (001) (010), in the acute angle  $p$   $o^1$  (001) (101). The plane of the optical axes is in  $g^1$  (010); the elongation is negative.

The density of the crystals, determined with 5·02 grms. of material, was found to be 5·290 at 15°.

## Phosphate and Arseniate of Bismuth.

**The Salt  $\text{BiPO}_4$ .**—I have crystallised this body by applying the method which gave me monetite, haidingerite, and other crystalline phosphates and arseniates (*Bull. Soc. Chim.*, vol. xxiv., p. 323, vol. xxvi., pp. 18, 24, 81, and 87). I dissolved in a large flask 15 grms. of the salt  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  and 7 grms. of the salt  $\text{HNa}_2\text{PO}_4 \cdot 12\text{H}_2\text{O}$  in concentrated nitric acid and a little water; I heated the solution on the water-bath, and allowed water to fall into it very slowly drop by drop. After some time the microscopic crystals were collected; they were about 0·15 m.m. long, and 0·05 m.m. thick.

When heated to redness, these crystals suffered no loss. On analysis they gave the following figures, leading to the formula  $\text{BiPO}_4$ :—

|                                          | Found. | Calculated. |
|------------------------------------------|--------|-------------|
| $\text{Bi}_2\text{O}_3$ (by diff.) .. .. | 76·79  | 76·60       |
| $\text{P}_2\text{O}_5$ .. ..             | 23·21  | 23·40       |
|                                          | 100·00 | 100·00      |

The crystals are limpid and brilliant, and take the form of small monoclinic prisms, elongated along the zone  $h^1m$  (100) (110), and terminated by hemipyramids. The face  $h^1$  (100) is often the dominant face. On this face the extinction is longitudinal. The plane of the optical axes is perpendicular to  $h^1$  (100). On  $m$  (110) extinction occurs at an angle of about  $9^\circ$  with the elongation in the obtuse angle formed by the edge of the pyramid and of the prism, and the edge  $h^1m$  (100) (110). The elongation is negative. The density of the crystals, determined on 7.55 grms. of material, was found to be 6.323 at  $15^\circ$ .

*The Salt  $\text{BiAsO}_4$ .*—This body can be obtained in the crystalline form if, in the corresponding preparation of the phosphate, we substitute for the disodic phosphate the equivalent amount of disodic arseniate or of arsenic acid.

The crystals are 0.1 m.m. in length and 0.036 m.m. in thickness. They are anhydrous, and analysis gives the following figures leading to the formula  $\text{BiAsO}_4$  :—

|                                            | Found. | Calculated. |
|--------------------------------------------|--------|-------------|
| $\text{Bi}_2\text{O}_3$ .. .. .            | 66.80  | 66.91       |
| $\text{As}_2\text{O}_3$ (by diff.) .. .. . | 33.20  | 33.09       |
|                                            | 100.00 | 100.00      |

This arseniate is in monoclinic prisms terminated by hemipyramids. They resemble the corresponding phosphate. The face  $h^1$  (100) is wanting. Extinction on  $m$  (110) takes place as in the corresponding phosphate. The elongation is negative.

The density of the crystals, determined with 7.80 grms. of material, was found to be 7.142 at  $15^\circ$ .—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 14.

### LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JANUARY 30TH, 1904.

By SIR WILLIAM CROOKES, F.R.S.,  
and  
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, ESQ., M.Inst.C.E.,  
*Water Examiner, Metropolitan Water Act, 1871.*

London, February 10th, 1904.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 211 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Jan. 1st to Jan. 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 211 samples examined by us during the month, all were clear, bright, and well filtered.

The rainfall at Oxford shows a considerable excess for January. The actual amount measured being 3.10 inches, of which 1.3 inches fell on the last two days. The average rainfall for the month is 2.08 inches; so we have an excess of 1.02 inches, equal to 49.0 per cent above the thirty-five years' average.

Our bacteriological examinations of 379 samples taken during last month have given the results recorded in the

following table. Besides these samples we have examined 390 others from special wells, standpipes, &c., making 769 samples in all :—

|                                                                                                                | Microbes<br>per c.c. |
|----------------------------------------------------------------------------------------------------------------|----------------------|
| New River, unfiltered (mean of 26 samples) ..                                                                  | 310                  |
| New River, filtered (mean of 73 samples) ..                                                                    | 16                   |
| Thames, unfiltered (mean of 26 samples) ..                                                                     | 8797                 |
| Thames-derived water from the clear-water wells of eight Thames-derived supplies (mean of 203 samples) .. .. . | 21                   |
| Ditto ditto .. .. .                                                                                            | highest 154          |
| Ditto ditto .. .. .                                                                                            | lowest 0             |
| River Lea, unfiltered (mean of 26 samples) ..                                                                  | 341                  |
| River Lea, from the East London Water Company's clear-water wells (mean of 25 samples) ..                      | 14                   |

Of the 301 daily samples taken from the general filter wells of the Metropolitan Water Companies, nineteen samples, or 6.3 per cent, were sterile. Ten samples, or 3.3 per cent, contained more than 100 microbes, and of these, only one sample contained more than 150 microbes per c.c., viz., 154. The ten excess samples contained an average of 119 microbes per c.c. In December thirty-one excess samples contained an average of 156 microbes per c.c. Thus the character of the general supply as regards bacterial impurity has improved greatly during the last month.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

### PRECIPITATION AND SEPARATION BY WEAK ORGANIC BASES.\*

By E. T. ALLEN.

(Continued from p. 78).

#### Saponification of Methyl Acetate by N/10 Hydrochloric Acid at $40^\circ\text{C}$ .

25 c.c. acid and 1 c.c. methyl acetate were used in each test. 1 c.c. acid = 0.00374 gm. hydrochloric acid, as determined by sodium carbonate. The process was carried out in sealed tubes as described above :—

$a_0$  = 1 c.c. of the original mixture in terms of N/20 soda = 2.10.

$a_\infty$  = 1 c.c. of the mixture when saponification was complete = 9.50.

$a = a_\infty - a_0 = 7.4$  = the original mass of methyl acetate in terms of N/20 soda.

Then the saponification constant for a reaction of the first order =  $K = \frac{1}{t} \log_e \frac{a}{a-x}$ .

Here, of course,  $t$  = the time, in this case measured in minutes, and  $x$  = quantity of methyl acetate changed, or acid liberated in the time  $t$ , again in terms of the soda solution. The Briggs logarithms were used in the calculation.

|                      |              |                  |
|----------------------|--------------|------------------|
| $t_1 = 131\text{ m}$ | $x_1 = 2.25$ | $K_1 = 0.001204$ |
| $t_2 = 235\text{ m}$ | $x_2 = 3.55$ | $K_2 = 0.001207$ |
| $t_3 = 324\text{ m}$ | $x_3 = 4.33$ | $K_3 = 0.001180$ |
| $t_4 = 363\text{ m}$ | $x_4 = 4.70$ | $K_4 = 0.001206$ |

Average = 0.00120

Corrected for volume, since 1 c.c. methyl acetate was added to the 25 c.c. acid,  $K = 0.00120 \times \frac{26}{25}$ .

Corrected for concentration, since the acid was not exactly N/10,  $K = 0.00120 \times \frac{26}{25} \times \frac{3645}{25.3740} = 0.001216 = 1.22 \times 10^{-3}$ .

\* From the *Journal of the American Chemical Society*, xxv., No. 4.

Walker (*Zeit. Phys. Chem.*, iv., 324) found for the velocity constant of the same reaction at 25° C.,  $K = 0.00315$ , using normal hydrochloric acid. Ley (*Zeit. Phys. Chem.*, xxx., 230) found at 99.7° C.,  $K = 0.00158$  as the average for N/500 hydrochloric acid, and 0.00307, average for N/250 hydrochloric acid.

If these constants are made comparable by multiplying them by their corresponding concentrations, we have—

$$\begin{aligned} kv &= 0.00315 \text{ at } 25^\circ \text{ C.} \text{---Walker.} \\ &= 0.01216 \text{ at } 40^\circ \text{ C.} \text{---Allen.} \\ &= 0.782 \text{ (average) at } 99.7^\circ \text{ C.} \text{---Ley.} \end{aligned}$$

Strictly these quantities should, of course, be multiplied by 2.3, because the natural logarithms were not used in the calculations.

Van't Hoff has shown that we may get some light on the change of reaction velocity with temperature if we resolve the equation  $\frac{d \log_e K}{dT} = \frac{q}{RT^2}$  (where  $K$  = the

equilibrium constant,  $\frac{k}{k'}$ , of any reaction,  $T$  = the absolute temperature, and  $q$  = the heat of reaction) into two others, viz.,  $\frac{d \log_e k}{dT} = \frac{A}{T^2} + B$ , and  $\frac{d \log_e k'}{dT} = \frac{A'}{T^2} + B$ .  $B$  is unknown, but often seems to be negligible, in which case, assuming also that  $q$  is constant, we have  $\log_e k = -\frac{A}{T} + C$  or  $\log_e \frac{k_2}{k_1} = A \left( \frac{T_2 - T_1}{T_1 T_2} \right)$ . By the aid of this equation, we may correlate the various values of  $k$  found at different temperatures.

Substituting for  $k_2$ ,  $k_1$ ,  $t_2$  and  $t_1$ , 0.01216, 0.00315, 298 and 313 respectively,  $A = 8397$ .

Substituting 0.782, 0.01216, 372.7, and 313,  $A = 8132$ . Considering the difference in dissociation of the different concentrations of acid, the slight variation of  $q$ , &c., the agreement seems satisfactory.  $K_T$  (the saponification constant of methyl acetate by hydrochloric acid) may thus be approximately reckoned from the equation—

$$K_T = \frac{K_{298}}{V} e^{8264 \left[ \frac{T - 298}{298 T} \right]}.$$

#### Saponification of Methyl Acetate by Aniline Hydrochloride at 40° C.

$a_0 + 1.81$  = acid in 1 c.c. mixture, at the beginning, in terms of N/20 soda.

$a_{\infty} = 9.30$  = free acid in 1 c.c. mixture after one month in the thermostat at 40° C.

$a = a_{\infty} - a_0 = 7.49$  total acid freed by saponification in 1 c.c. mixture, equivalent to the original mass of the methyl acetate in terms of soda.

$$K = \frac{1}{t} \log_e \frac{a}{a-x}.$$

$$t_1 = 1330 \text{ m} \quad x_1 = \begin{cases} 2.35 \\ 2.31 \\ 2.32 \end{cases}$$

$$2.33 - 1.81 = 0.52$$

$$t_2 = 2450 \text{ m} \quad x_2 = \begin{cases} 2.80 \\ 2.85 \\ 2.81 \end{cases}$$

$$2.83 - 1.81 = 1.02$$

$$t_3 = 4290 \text{ m} \quad x_3 = \begin{cases} 3.61 \\ 3.60 \end{cases}$$

$$3.61 - 1.81 = 1.80$$

$$t_4 = 5683 \text{ m} \quad x_4 = \begin{cases} 4.20 \\ 4.23 \end{cases}$$

$$4.22 - 1.81 = 2.41$$

$$1. K = 2.35 \times 10^{-4} \times \frac{26}{25} = 2.45 \times 10^{-4}.$$

$$2. K = 2.60 \times 10^{-4} \times \frac{26}{25} = 2.70 \times 10^{-4}.$$

$$3. K = 2.78 \times 10^{-4} \times \frac{26}{25} = 2.89 \times 10^{-4}.$$

$$4. K = 2.97 \times 10^{-4} \times \frac{26}{25} = 3.09 \times 10^{-4}.$$

Now if it be remembered that the rate of reaction depends on the concentration of free hydrochloric acid, it will be seen that the percentage of aniline salt which is hydrolysed may be obtained by dividing these results by the constant for hydrochloric acid at the same dilution:—

$$2.45 \times 10^{-4} \div 1.22 \times 10^{-4} = 2.0 \text{ per cent.}$$

$$2.70 \quad \text{"} \quad \text{"} \quad \text{"} \quad 2.2 \quad \text{"}$$

$$2.89 \quad \text{"} \quad \text{"} \quad \text{"} \quad 2.4 \quad \text{"}$$

$$3.09 \quad \text{"} \quad \text{"} \quad \text{"} \quad 2.5 \quad \text{"}$$

As there is always a constant ratio between the undecomposed part of the salt, multiplied by the volume in which it is contained, and the product of the decomposed parts, we have the well-known equation  $\frac{x}{V(1-x)} = C$ ;

where  $x$  is the percentage hydrolysed,  $V$  is the volume in which 1 grm.-molecule is dissolved, and  $C$  is the hydrolytic constant. If we take the average of  $x$  as 2.3,  $C = 5.4 \times 10^{-4}$ , or if 2 is more nearly correct,  $C = 4.1 \times 10^{-4}$ .

The strength of aniline has previously been determined by two different observers. Bredig (*Zeit. Phys. Chem.*, xiii., 322), using the conductivity method, concluded that 2.63 per cent of aniline hydrochloride was decomposed in N/32 solution at 25° C., while Walker (*Journ. Chem. Soc.*, lxvii., 582), by the use of the sugar-inversion method, obtained the value 4.5 per cent at 60° C. in N/30 solution.

If we calculate these results to N/10 dilution by the use of the equation  $C = \frac{x^2}{v(1-x)}$ , we have—

$$1.48 \text{ per cent hydrolysed at } 25^\circ \text{ C.} \text{---Bredig.}$$

$$2.00 \quad \text{"} \quad \text{"} \quad 40^\circ \text{ C.} \text{---Allen.}$$

$$2.63 \quad \text{"} \quad \text{"} \quad 60^\circ \text{ C.} \text{---Walker.}$$

These results indicate a nearly regular increase in hydrolysis with increasing temperature. Regarding this point not much is known, but Madsen's results indicate that the increase may be very considerable (*Zeit. Phys. Chem.*, xxxvi., 294).

(To be continued).

#### ON THE COLORIMETRIC DETERMINATION OF SMALL QUANTITIES OF PHOSPHORIC ACID AND OF SILICA.\*

By F. P. VEITCH.

(Continued from p. 74).

##### Effect of varying the Amount of Nitric Acid present.

WOODMAN and Cayvan (*Journ. Am. Chem. Soc.*, xxiii., 96) found that the depth of colour and the rapidity with which it developed depended to a certain extent on the amount of reagents present in the solution, and further state that the best results were obtained with 4 c.c. of ammonium molybdate solution (50 grms. of the salt per litre) and 2 c.c. of nitric acid (sp. gr. 1.07) per 50 c.c. of the solution to be examined.

It appeared advisable to make an experimental study of these points in order to obtain data on the allowable variations in amounts of added reagents. To this end solutions containing different amounts of nitric acid, and also different amounts of standard phosphate solution, were compared with solutions containing 2 c.c. of nitric acid and 4 c.c. of molybdate. The results follow:—

\* From the *Journal of the American Chemical Society*, xxv., No. 2.



TABLE V.—Effect of varying the Quantity of Nitric Acid added.

| HNO <sub>3</sub> + Na <sub>2</sub> HPO <sub>4</sub> + Molybdate = Na <sub>2</sub> HPO <sub>4</sub> + HNO <sub>3</sub> + Molybdate. |      |      |      |      |      |
|------------------------------------------------------------------------------------------------------------------------------------|------|------|------|------|------|
| C.c.                                                                                                                               | C.c. | C.c. | C.c. | C.c. | C.c. |
| 1                                                                                                                                  | 1    | 4    | 0.83 | 2    | 4    |
| 2                                                                                                                                  | 1    | 4    | 1.04 | 2    | 4    |
| 3                                                                                                                                  | 1    | 4    | 1.10 | 2    | 4    |
| 4                                                                                                                                  | 1    | 4    | 1.15 | 2    | 4    |
| 5                                                                                                                                  | 1    | 4    | 1.24 | 2    | 4    |
| 6                                                                                                                                  | 1    | 4    | 0.98 | 2    | 4    |
| 7                                                                                                                                  | 1    | 4    | 0.97 | 2    | 4    |
| 8                                                                                                                                  | 1    | 4    | 0.88 | 2    | 4    |
| 10                                                                                                                                 | 1    | 4    | 0.84 | 2    | 4    |

The intensity of the colour is sharply and regularly affected by the amount of nitric acid present, and it is of particular interest to note that there is a maximum point at which the colour is most intense; with less acid or with more acid present the intensity of colour is considerably reduced, and it is evident that to obtain strictly comparable results the standard and the solution to be read must contain, in a given volume, sensibly the same amount of acid.

Possibly these results are also of interest in throwing light on the precipitation of ammonium phosphomolybdate, and may explain the results sometimes obtained. It is a well-known fact that the colour of the precipitate varies with the amount of acid present from a light lemon-yellow to a deep orange, and the speed with which the precipitate is formed also varies with the amount of nitric acid present. Under the best conditions this precipitation takes place rapidly, and is complete; the supernatant fluid is practically as colourless as water, even when it contains a considerable amount of iron salt. On the other hand, under some other condition the precipitate forms slowly, is "muddy," filters and washes slowly, and shows a decided tendency to run through the filter, while the filtrate generally has a decided colour, due apparently to the ammonium phosphomolybdate still in solution.

The above results show that the presence of 5 c.c. of nitric acid (sp. gr. 1.07) per 50 c.c. of solution gives the most intense colour, and this amount has been adopted for this work. Whether or not this relation gives the optimum condition for the precipitation of ammonium phosphomolybdate, remains to be investigated.

#### Effect of varying the Quantity of Molybdate.

The effect of varying the quantity of molybdate was studied in the same way. The standard and the solution to be read were identical in all particulars except that in the standard the amount of molybdate present in 50 c.c. was always 4 c.c., while in the other solution the amount of molybdate varied.

TABLE VI.—Effect of varying the Quantity of Molybdate added.

| Molybdate + Na <sub>2</sub> HPO <sub>4</sub> + HNO <sub>3</sub> = Na <sub>2</sub> HPO <sub>4</sub> + Molybdate + HNO <sub>3</sub> . |      |      |      |      |      |
|-------------------------------------------------------------------------------------------------------------------------------------|------|------|------|------|------|
| C.c.                                                                                                                                | C.c. | C.c. | C.c. | C.c. | C.c. |
| 2                                                                                                                                   | 1    | 5    | 0.85 | 4    | 5    |
| 4                                                                                                                                   | 1    | 5    | 1.00 | 4    | 5    |
| 6                                                                                                                                   | 1    | 5    | 1.15 | 4    | 5    |
| 8                                                                                                                                   | 1    | 5    | 0.97 | 4    | 5    |
| 12                                                                                                                                  | 1    | 5    | 0.96 | 4    | 5    |

The results show considerable differences due to varying the amount of molybdate. These differences are not as great nor is the maximum point as well defined as with different amounts of nitric acid, and there seems to be no reason for changing the amount of molybdate adopted by Woodman and Cayvan as giving the most satisfactory results.

From the foregoing results it appears reasonably certain that in order to obtain reliable and comparable results, the standard and the unknown solution must always contain the same quantities of nitric acid and of molybdate, and be at the same temperature. Should different workers use different amounts of reagent, their results could not be strictly comparable.

#### Effect of Filter-paper.

In the experiments on the removal of silica, and also in some of those on the loss of phosphoric acid on evaporation of the solution, it was observed that the readings were higher than they should have been. As all the work was done in platinum dishes, the only point where materia could get in was where the solutions were filtered to remove silica. In order to determine if anything is taken up from the filter, solutions of various strength were passed through 7 c.m. S. and S. No. 590 paper with the following results:—

| Parts per million before filtering. | Parts per million after filtering. | Gain, parts per million. |
|-------------------------------------|------------------------------------|--------------------------|
| 1                                   | 1.16                               | 0.16                     |
| 1                                   | 1.14                               | 0.14                     |
| 2                                   | 2.12                               | 0.12                     |
| 2                                   | 2.17                               | 0.17                     |
| 5                                   | 5.01                               | 0.01                     |
| 5.5                                 | 5.50                               | 0.00                     |

The filters were washed until filtrates had a volume of 50 c.c. each. The results indicate that in passing through the filter these nitric acid solutions of phosphates have taken up silica, phosphoric acid, or organic matter equal to an average reading of 0.15 part per million in four cases, while in two experiments there is no apparent gain. It seems probable that in these latter cases the amount taken up from the paper is masked by incomplete removal of the phosphoric acid from the paper, in the volume of wash-water used. Beyond a doubt filtration introduces a plus error, which may only be apparent in very dilute solutions. Results given in this paper have been corrected in accordance with the above data.

#### Removal of Silica.

As it is necessary to remove silica from the solution before reading, it was thought that this might be most expeditiously and certainly accomplished by treatment with hydrofluoric acid.

Repeated trials, however, showed that this is not true. While the silica was completely removed finally, it was found that it was necessary to remove all hydrofluoric acid before standardising, as the smallest quantity of fluorides prevented completely the development of the colour. As at least three evaporations with nitric acid were required to accomplish the removal of hydrofluoric acid, the standard procedure of evaporation with nitric acid and drying at 100° for two hours was returned to.

The writer's experience confirms the statement of Woodman and Cayvan, that a single evaporation removes completely the silica from the solution in those cases where lime and magnesia salts are absent, but where lime and magnesia salts are present in considerable quantities, the amount of silica remaining in solution after one evaporation and filtration is too great to be neglected.

For the study of these points a solution of sodium silicate was prepared which, by gravimetric analysis, gave from first evaporation and filtration, 266 parts per million SiO<sub>2</sub>; second evaporation and filtration, 2 parts per million SiO<sub>2</sub>; third evaporation and filtration, 0 part per million SiO<sub>2</sub>; total 268 parts per million SiO<sub>2</sub>.

The filtrate from the third evaporation contained no silica by the colorimetric method. 250 c.c. of this silicate solution were transferred and made up to 1 litre, with the view of obtaining a solution containing about as much dissolved silica as is found in many soil extracts.

By calculation from the above determinations this contained 67 parts SiO<sub>2</sub> per million; first evaporation and filtration removed 63 parts SiO<sub>2</sub> per million; second evaporation and filtration removed 2 parts SiO<sub>2</sub> per million; total recovered, 65 parts SiO<sub>2</sub> per million.

By the colorimetric method the solution contained 65 parts SiO<sub>2</sub> per million.

The filtrate from the first evaporation contained, colorimetrically, less than 0.05 part per million, average of four

determinations. The filtrate from the second evaporation contained, colorimetrically, less than 0.1 part per million. The filtrate from the third evaporation contained, colorimetrically, less than 0.1 part per million.

In order to determine the effect of the presence of lime and magnesia salts, a solution containing 25 m.grms. of calcium nitrate and 25 m.grms. of magnesia was added to portions of silicate solution and silica determined colorimetrically as well as gravimetrically.

Gravimetrically obtained:—First evaporation and filtration removed 63 parts SiO<sub>2</sub> per million; second evaporation and filtration removed 5 parts SiO<sub>2</sub> per million; total 68.

Colorimetrically obtained:—67 parts SiO<sub>2</sub> per million; calculated, 67 parts SiO<sub>2</sub> per million.

Filtrate from the first evaporation gave, colorimetrically, 1.42 parts SiO<sub>2</sub> per million. Filtrate from the second evaporation gave, colorimetrically, 0.1 part SiO<sub>2</sub> per million. Filtrate from the third evaporation gave, colorimetrically, less than 0.1 part SiO<sub>2</sub> per million.

From these results it appears that in the absence of lime and magnesia salts the silica left in most soil solutions after one evaporation may be neglected, but in the presence of these salts two evaporations are necessary.

(To be continued).

## WHAT PHYSICAL CHEMISTRY HAS DONE FOR CHEMISTRY.\*

By HARRY C. JONES.

PHYSICAL chemistry has furnished us with several generalisations which have greatly modified our methods of dealing with chemical phenomena. Among these may be cited the theory of electrolytic dissociation, the law of mass action, Faraday's law, the basis of chemical valence, and the phase rule.

Of these generalisations time will permit us to consider only one or two, and we can only make a few applications of the most important of them all—the theory of electrolytic dissociation.

A word or two in reference to the origin of the theory.

**Osmotic Pressure.**—When a solution of any substance is brought in contact with the pure solvent, or with a solution of different concentration, at the surface of contact of the two solutions there exists a pressure which tends to drive the dissolved substance from the region of greater to the region of lesser concentration. This is known as osmotic pressure. The first to measure the magnitude of this pressure was the botanist, Wilhelm Pfeffer, who found that salts gave a greater osmotic pressure, for equivalent concentrations, than solutions of substances like cane-sugar.

**Origin of the Theory of Electrolytic Dissociation.**—It remained for van't Hoff to point out that the electrolytes in general—acids, bases, and salts—exerted a greater osmotic pressure than the non-electrolytes. He also showed that osmotic pressure is a property which depends only upon the number of parts of the dissolved substance in a given volume of the solvent, and not upon the nature of the parts. Since osmotic pressure depends only upon the ratio between the number of parts of the dissolved substance and the number of parts of the solvent, and since electrolytes exert an osmotic pressure that is greater than can be accounted for on the basis of the existence of these substances in the molecular condition, we are forced to the conclusion that the molecules of electrolytes are broken down in solution into parts. These part molecules are called *ions*.

I cannot not take up here the evidence bearing upon the theory of electrolytic dissociation, since it would lead much too far. Suffice it to say that the experimental evidence, as far as it has been established in a trustworthy

manner, is practically unanimously in favour of the theory, and the amount of such evidence already at hand is simply overwhelming. The theory of electrolytic dissociation is as well established as most of our laws in science, and is to-day almost universally accepted by the leading chemists and physical chemists throughout the world.

**Neutralisation of Acids and Bases.**—Let us now see what the theory has done for chemistry. Here again we can take up only a few of the many applications of this most important generalisation. Take a class of chemical reactions that are more or less familiar to every one—the neutralisation of acids and bases. It has long been known that when an acid is brought in contact with a base, both are neutralised, and when the solution is evaporated a salt is obtained. Since a different salt was obtained with every acid and base employed, we had as many new phases of the problem of neutralisation as we had acids and bases to react.

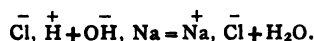
This whole subject has been beautifully unified and simplified by means of our theory. Take the neutralisation of hydrochloric acid with sodium hydroxide. Hydrochloric acid in dilute solution is a mixture of hydrogen ions and chlorine ions:—



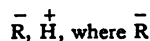
Sodium hydroxide in dilute solution is a mixture of sodium ions and hydroxyl ions:—



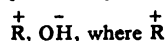
When these solutions are brought together we have:—



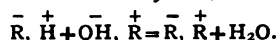
What takes place and all that takes place in the above case is the union of the hydrogen ion of the acid with the hydroxyl ion of the base, forming a molecule of water. The anion of the acid, chlorine, and the cation of the base, sodium, remain after the process of neutralisation in exactly the same condition as before neutralisation took place, and a salt is formed only when the solvent which dissociated the salt into its ions is removed. We may now discuss the subject of neutralisation in general. We may represent any acid by:—



is the anion, which differs in composition for every acid, and the cation hydrogen which is common to every acid. Similarly, we may represent any base by:—



is the cation of the base. Its composition varying with every base, and hydroxyl is the anion common to all bases. When any base reacts with any acid, we have:—



Water is always formed, and the anion of the acid  $\overset{-}{\text{R}}$ , and the cation of the base  $\overset{+}{\text{R}}$ , remain in the solution after the process of neutralisation in exactly the same condition as before neutralisation took place. In order to obtain the salt the water which holds the ions apart must be removed.

Neutralisation, then, in terms of our theory consists in the formation of a molecule of water from a molecule of the acid and a molecule of the base, and in nothing else. One process of neutralisation is, therefore, exactly the same as any other process of neutralisation, regardless of the nature of the acid or the nature of the base involved. If this is true it is very important, since it refers all the processes of neutralisation to a common cause—the union of the hydrogen ion of the acid with the hydroxyl ion of the base.

(To be continued).

\* From the *Journal of the Franklin Institute*, December, 1903.

By means of the latter reaction, several new acetylated thioureas have been prepared, together with others (for example, acetylthiourea), which, although previously described, have not hitherto been obtained directly from the parent substance. The yields obtained with various bases and at different temperatures have also been studied.

20. "Resolution of  $\alpha\beta$ -Dihydroxybutyric Acid into its Optically Active Constituents." By ROBERT SELBY MORRELL and EDWARD KENNETH HANSON.

Of a series of salts of the active bases with  $\alpha\beta$ -dihydroxybutyric acid, derived from  $\alpha$ -crotonic acid, the quinidine salt is the only one which admits of resolution into its two active components, so that these can be separated by crystallisation from water. The quinidine *l*-salt is very sparingly soluble in this solvent, and from it the *l*- $\alpha\beta$ -dihydroxybutyric acid has been prepared. The free acid has the same melting point as the racemic substance, but it crystallises in six-sided plates and not in needles. Its specific rotation is  $13.5^\circ$ , this being numerically greater than that of *d*-glyceric acid, which has  $[\alpha]_D +2.14^\circ$  (Frankland and Frew, *Trans.*, 1891, lix., 96), and smaller than that of *l*- $\beta$ -hydroxybutyric acid ( $[\alpha]_D -24.8^\circ$ ) (MacKenzie, *Trans.*, 1903, lxxxi., 1402). The barium salts of the *d*- and *l*-acids have been prepared, and the specific rotation of the barium *l*-salt is greater than that of the free acid. Faber and Tollens (*Ber.*, 1899, xxxii., 2598) have described salts of a dihydroxybutyric acid, obtained, together with isosaccharic acid, by the action of lime water on oxycellulose. The rotation of this  $\alpha\beta$ -dihydroxybutyric acid is given as  $+13.7^\circ$ , and it is probable that this acid is the *d*-isomeride, corresponding with the *l*-modification obtained from  $\alpha$ -crotonic acid.

21. "Aromatic Compounds obtained from the Hydro-aromatic Series. (Part I.) The Action of Bromine on 3:5 Dichloro-1:1-dimethyl- $\Delta^2$ :4-dihydrobenzene." By ARTHUR WILLIAM CROSSLEY.

Dichlorodimethyldihydrobenzene, on treatment with two molecular proportions of bromine, yields dichlorotribromodimethyltetrahydrobenzene,  $C_8H_9Cl_2Br_3$ , crystallising in stout, prismatic needles and melting at  $118^\circ$ . When heated to a few degrees above its melting point, this substance rapidly evolves two molecular proportions of hydrogen bromide, giving rise to 3:5-dichloro-4-bromo-*o*-xylene, which crystallises in felted needles melting at  $100^\circ$ . The constitution of this aromatic compound has been definitely decided by synthesis.

If, however, dichlorodimethyldihydrobenzene is treated with only one molecular proportion of bromine, an unstable liquid results, which, on heating, loses hydrogen bromide, giving as the main products 3:5-dichloro-*o*-xylene and 3:5-dichloro-6-bromo-*o*-xylene (m. p.  $42^\circ$ ).

On further bromination, both these dichlorobromoxylenes give 3:5-dichloro-4:6-dibromo-*o*-xylene, crystallising from ethyl acetate in glistening needles (m. p.  $233^\circ$ ), but they differ markedly in their behaviour towards nitric acid.

Thus, 3:5-dichloro-4-bromo-*o*-xylene, on treatment with fuming nitric acid, gives 3:5-dichloro-4-bromo-6-nitro-*o*-xylene, and yields 3:5-dichloro-4-bromo-*o*-phthalic acid when oxidised with dilute nitric acid under pressure, whereas under similar conditions, 3:5-dichloro-6-bromo-*o*-xylene furnishes respectively 3:5-dichloro-4:6-dinitro-*o*-xylene and 3:5-dichloro-6-nitro-*o*-toluic acid.

22. "The action of Nitrogen Sulphide on Organic Substances." (Part I.) By FRANCIS ERNEST FRANCIS and OLIVER CHARLES MINTY DAVIS.

Nitrogen sulphide reacts readily with aromatic aldehydes. Benzaldehyde gives triphenylcyanidin (cyaphenin) and a small quantity of lophin, probably formed by the reduction of the former compound by the sulphur dioxide liberated in the reaction. *p*-Tolualdehyde reacts similarly, giving the corresponding tritolylcyanidin. Anisaldehyde, however, yields only very small quantities of *p*-trimethoxyphenylcyanidin, the main product of the reaction being a white, insoluble powder, which is at present under investigation.

23. "Dibenzoylchloroimide." By FREDERICK DANIEL CHATTAWAY.

The author points out that Stieglitz and Earle have overlooked his paper (*Proc.*, 1902, xviii., 165; *Chem. Centr.*, 1902, II., 359), communicated to the Society on June 18th, 1902, which contains a description of the preparation and properties of the diacylchloroimides,

including the dibenzoylchloroimide again described by these investigators (*Amer. Chem. Journ.*, 1903, xxx., 420). Stieglitz and Earle state that, owing to the ease with which the diacylchloroimides are hydrolysed, it is necessary to exclude water entirely in the preparation of these substances. This, however, is contrary to the author's experience, for the chloroimides are readily obtained in the presence of water in the manner indicated in the former paper (*Proc.*, *ibid.*).

Dibenzoylchloroimide, when pure, melts at  $80^\circ$ , or three degrees higher than the temperature given by Stieglitz and Earle.

The diacylchloroimides are readily hydrolysed, yielding the diacylimides and hypochlorous acid either when left in contact with water or when heated with this medium, but the reaction is reversible.

## PHYSICAL SOCIETY.

Annual General Meeting, February 12th, 1904.

Dr. R. T. GLAZEBROOK, F.R.S., President, in the Chair.

THE Report of the Council and the Report of the Treasurer were read by the Secretary.

The following Officers and Council were elected for the ensuing year:—

President—R. T. Glazebrook, D.Sc., F.R.S.

Vice-presidents—Those who have filled the Office of President, together with T. H. Blakesley, M.A.; C. Chree, D.Sc., F.R.S.; Prof. J. D. Everett, D.C.L., F.R.S., and J. Swinburne.

Secretaries—W. Watson, D.Sc., F.R.S., and W. R. Cooper, M.A.

Foreign Secretary—Prof. S. P. Thompson, D.Sc., F.R.S.

Treasurer—Prof. H. L. Callendar, M.A., F.R.S.

Librarian—W. Watson, D.Sc., F.R.S.

Other Members of Council—C. V. Boys, F.R.S.; W. Cassie, M.A.; W. B. Croft, M.A.; H. M. Elder, M.A.; Prof. J. Perry, D.Sc., F.R.S.; A. W. Porter, B.Sc.; W. A. Price, M.A.; Prof. F. T. Trouton, Sc.D., F.R.S.; W. C. D. Whetham, M.A., F.R.S.; S. A. F. White, M.A.

THE PRESIDENT then delivered an Address.

He said that during the last session the attention of the Society had been drawn, by more than one paper, to the study of geometrical optics. He wished now to deal with one or two matters connected with the theory of the microscope. Geometrical optics in its relation to instruments has been studied to great advantage abroad, but, of recent years, has been somewhat neglected in this country. The result is that only a small share in the recent advance in lens construction has been ours. In 1878 Abbe first called attention to the fact that the future perfection of the microscope as an optical instrument depended on the advance of the art of glass-making. With the glasses then at their disposal it was impossible for opticians to get rid of the secondary spectrum of their object-glasses which prevented the attainment of the highest perfection of the image. It was to this fact that the establishment of the firm of Schott and Company was due. Contrasting what is now possible, so far as achromatic correction is concerned, with what was possible say twenty years ago, the President drew attention to the Lick refractor of 56 feet focus, and pointed out that there is a difference of over 3 inches in the positions of the focus for D and H. It would now be possible to construct a lens of 60 feet focus, so that this difference would be reduced to about one-seventh of an inch. Turning to the microscope, and taking Abbe and his pupils as his guide, Dr. Glazebrook analysed the action of the parts of the instrument in order to see if further improvement was possible. It was shown that by the use of two lenses of appreciable focal length, say 10 m.m. each, we can, by placing them at a distance, say 100 m.m. apart, get an equivalent lens of shorter

focus, 1 m.m. in the case in point, and of correspondingly higher power. The advantage of this is obvious; if for no other reason, because of the greater ease of working large lenses. If we imagine a plane perpendicular to the axis of a microscope dividing the object-glass into two parts so that the divergent pencil which falls upon the lens is a parallel pencil when it crosses the plane, we see that a microscope may be considered as a telescope with a short focus lens put in front and used to view an image in the principal focus of that lens. We owe to Abbe the introduction of the term numerical aperture, by which is meant the value of the quantity  $\mu \sin \alpha$ , where  $\mu$  is the refractive index of the medium in which the object is placed, and  $2\alpha$  the vertical angle of the cone subtended at the object-glass by the point in which the axis of the instrument meets the object. Let us suppose that an object is on the stage and viewed by transmitted light, and to simplify matters let us suppose the source of light at some distance. Then, according to Abbe and his followers, in considering the image formed in the focal plane of the eyepiece we are not to start from the object as a self-luminous source, and consider where the image of such a source would be if formed by the laws of geometrical optics; we are told to start from the source itself, to consider its image formed in the focal plane of the object-glass, and to treat this image as the source of light in the microscope-tube, from which arises the image we see. If the object be small the focal image will be modified by diffraction by the object; and according to Abbe it is on the nature of the diffraction images and the number of them which are formed, that the definition depends. Suppose the microscope has been focussed on some object on the stage and that this object has been removed; the parallel rays from the source are brought to a focus in the focal plane of the object-glass, forming there a circular patch of light; from each point of this rays diverge and, reaching the eye, produce the sensation of a uniform luminous field. Now let the field in the focal plane be limited by a diaphragm pierced with a series of small apertures. The distribution of light in the focal plane of the eye-lens will be no longer uniform, and we shall see there the diffraction-pattern formed by the apertures. Instead, however, of producing a variable distribution in the focal plane of the object-glass by means of diaphragms, we can do it by means of the diffraction effects of small objects on the stage. Thus, if we put on the stage a grating consisting of a series of equidistant lines, then taking homogeneous light, a series of narrow bands of light, the diffraction images of the source, will be produced in the focal plane with darkness between them. On the assumption that the microscope satisfies the sine condition, the image in the view-plane is the ordinary geometrical image of the grating, a series of uniformly-spaced bright and dark bands. On the distribution of light in the interspaces between the maxima of brightness, the question of resolving power depends. If we modify the number of spectra in the focal plane we modify the image, and this has been done in an ingenious way in some of the experiments arranged by Prof. Abbe's pupils to illustrate his theory. If we cut out all but the central image, the view-field is uniform, no structure is visible; if we allow the first image on either side of the central one to become effective, the bands appear in the field in their proper positions and so on. It is said to be the fundamental result of Abbe's theory that the object (the grating) can be fully resolved if one diffraction image is formed on either side of the central one. It follows from this, that a grating is resolvable if the space between the lines is not less than the result found by dividing the wave-length of light by the numerical aperture. Dr. Glazebrook pointed out that although, by the reasoning he had outlined, the truth of this result could be established, it did not seem to him to prove it. In order to decide whether a grating can be resolved, we must establish the law of variation of intensity in the view-plane, and then consider whether these variations are such that they can be detected by the eye. This has been done by Lord

Rayleigh. The images formed in a microscope are, like all other images, produced by interference; in considering resolving power, we have to consider diffraction effects, it is true, but the diffraction which concerns us mainly is that due to the aperture of the object-glass, and only indirectly to that due to the object viewed. The size of the object on the stage only affects in a secondary manner the method in which the image is formed. It is not necessary, if we know completely the distribution of light over the stage, to go back to the source in a consideration of the problem. Given the distribution over the stage, both in amplitude and phase, we are potentially able to determine that in the view-plane without reference to the source. In the case of a grating illuminated by plane waves, their plane being parallel to that of the grating, we have to consider the effect due to a series of equidistant lines of light. These differ, however, from a series of independent equidistant linear sources, in that the phases of the various sources are the same, and we have therefore to remember that interference will take place between the light from the different lines, while in the latter case there is no relation between the phases, and we can calculate the intensity due to each source separately, and superpose the whole. The method adopted by Lord Rayleigh for the solution of the problem which is presented when a narrow double line in a spectrum is viewed through a telescope, has been applied by him to the microscope (*Phil. Mag.*, Aug., 1896). Dealing with two bright lines and a rectangular aperture, he has shown that, if a variation in intensity of 10 per cent can be detected in the view-plane, then, if the sources are independent, they can be resolved if the distance between them is half that given by Abbe's theory. Lord Rayleigh next considers the effect of a large number of equispaced independent sources, and shows that the view-field has a periodic structure, but that the amount of discontinuity gets less as the sources get nearer together. In the case of a grating with plane-waves incident normally, it is shown that the intensity can be expressed as the sum of a series of periodic terms, each of which corresponds to the effect of two of Abbe's lateral spectra, and that no resolution is possible unless at least the first spectrum on each side is effective. The field in the view-plane corresponds to a series of bright bands at the points corresponding to the geometrical images of the grating; midway between these are a series of less bright bands, while between each pair of these bright bands is an absolutely dark space.

It appears that, while Abbe's theory of microscopic vision is undoubtedly correct in that a small object or objects on the stage produce diffraction patterns in the focal plane of the object-glass, and the illumination in the view-plane can be inferred from these diffraction images, still his method of regarding the question is not the only possible one, neither is it necessary to go back to the original source in order to calculate the illumination. Moreover, we are certain to arrive at erroneous consequences if we apply results obtained from the case of a grating to a case such as that of a small aperture or a small obstacle; the diffraction patterns would be different, and the conditions of resolution different also.

Mr. Beck exhibited a series of microscopes in which diffraction-gratings on the stages were viewed with different arrangements of slits placed in the focal planes of the object-glasses, which illustrated the various points of Abbe's theory.

**Preparation of Primary Alcohols by means of the Corresponding Amides.**—L. Bouveault and G. Blanc. —The authors obtain, by means of sodium ethylic alcohol and the caproic, pelargonic, and phenylacetic amides, the corresponding alcohols:—(1) Normal hexylic alcohol,  $\text{CH}_3(\text{CH}_2)_4\text{CH}_2\text{OH}$ , boiling at  $156^\circ$ ; (2) normal nonylic alcohol,  $\text{CH}_3(\text{CH}_2)_7\text{CH}_2\text{OH}$ , boiling at  $215^\circ$ ; (3) phenylethyl alcohol,  $\text{C}_6\text{H}_5\text{CH}_2\text{CH}_2\text{OH}$ , boiling at  $212-215^\circ$ . These examples show that this reaction is general. —*Comptes Rendus*, cxxviii., No. 3.

## NOTICES OF BOOKS

*The Manuring of Market Garden Crops.* By BERNARD DYER, D.Sc., F.I.C., and F. W. E. SHRIVELL, F.L.S. Reprinted from the *Journal of the Royal Horticultural Society*, Vol. xxvii., Part 4. London: Vinton and Co., Ltd. 1903. Pp. 120.

THE experiments carried out by the authors have now reached their tenth year. This pamphlet contains the records of the first eight years, and, to a partial extent, those of the ninth year.

The work done resolves itself very largely into an enquiry as to whether or not the large quantities of purchased dung now used by the market gardeners are being used to the greatest economical advantage.

It is well known that market garden crops are manured more heavily than almost any others, and most of the manures used are chiefly nitrogenous, and the market gardener is too often not aware that by neglecting to supply the phosphates necessary to balance the nitrogen in them, he is preventing them from doing their full work.

The experiments which have been carried out at the Hallow Experimental Station, Golden Green, indicate over and over again that heavy dunging for most crops is wasteful and unprofitable, and that a much lighter dressing of dung, supplemented by chemical fertilisers, is the best application. The experiments cover all kinds of market garden produce, and the pamphlet is fully illustrated with photographs showing the different yields obtained from dung alone, and dung with chemical fertilisers, or the latter alone.

*An Elementary Text-book of Metallurgy.* By A. HUMBOLDT SEXTON, F.I.C., F.C.S., &c. Third Edition, thoroughly Revised and Enlarged. With numerous Illustrations. London: Charles Griffin and Co., Ltd. 1903. Pp. 263.

THE first edition of this text-book was published in 1894; it has evidently been well received, as we have now before us the third edition. This addition has been revised, and though the scope and plan of the book have been unchanged, there have been a few additions and many minor alterations.

Beginning as it does with the nature of metallurgy and the properties of metals, this book is specially adapted for the use of students. By its study they will acquire a good grounding in the science, and will the more easily understand the special methods and processes they intend to follow in the future.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

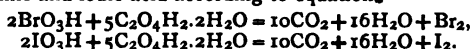
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 3, January 18, 1904.

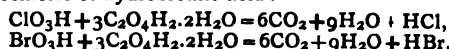
**Zinc Peroxides.**—M. de Forcrand.—The author makes a series of experiments on the peroxides of zinc. He proves the existence of a compound  $\text{ZnO}_{1.75} + \text{H}_2\text{O}$  or  $\text{Zn}_4\text{O}_7 + 4\text{H}_2\text{O}$ , which corresponds to one limit; then a very unstable compound  $\text{ZnO}_{1.98} + 2$  or  $2.5\text{H}_2\text{O}$ ; the degree of oxidation of this compound approaches very near to  $\text{ZnO}_2$ . Finally, by the action of heat on these former substances he isolates  $\text{ZnO}_{1.66} + \text{H}_2\text{O}$  or  $\text{Zn}_3\text{O}_5 + 3\text{H}_2\text{O}$ , and  $\text{ZnO}_{1.66} + 0.66\text{H}_2\text{O}$  or  $\text{Zn}_3\text{O}_5 + 2\text{H}_2\text{O}$ . At  $190^\circ$  and  $210^\circ$  respectively these two compounds suddenly decompose, giving an almost anhydrous protoxide without intermediate products.

**Method of Separation of Aluminium and Iron by the Use of Formic Acid.**—A. Leclère.—The author succeeds in separating aluminium and iron very exactly by the precipitation of the aluminium in the form of basic formate. For example, using a dilute solution containing a slight excess of sulphuric acid, first the iron is reduced to a protoxide salt. For this purpose it is best to use ammonium hyposulphite, as no residue is left in the precipitates. Ammonium formate is then added, and the whole kept boiling. The iron is kept in the form of protoxide, whilst the aluminium is precipitated gradually, not in the state of sulphite, but of basic formate mixed with a little sulphur. When drying the precipitate nitric acid should be added to drive off the formic acid and to prevent the presence of a residue of carbon in the calcined alumina. The iron can be precipitated by the addition of sulphuretted hydrogen to the liquid filtrate. The sulphide is produced.

**Estimation of Chlorates, Bromates, and Iodates.**—Léon Debourdeaux.—The estimation of chloric, bromic, and iodic acids can be made by determining the proportion of the halogen which is contained in the compound after it has been transformed by calcination into the alkaline or argentic chloride, bromide, or iodide. This estimation can be effected by a volumetric method analogous to that used for the estimation of nitrates, and founded on the following facts:—1. Oxalic acid is not destroyed at boiling-point by a solution containing less than 20 c.c. of concentrated sulphuric acid per 100 c.c. 2. Oxalic acid in a liquid containing 12 c.c. of concentrated sulphuric acid per 100 c.c. is destroyed by chloric acid according to equation  $\text{ClO}_3\text{H} + 2\text{C}_2\text{H}_4\text{H}_2.2\text{H}_2\text{O} = 4\text{CO}_2 + 6\text{H}_2\text{O} + \text{ClOH}$ , and by bromic and iodic acid according to equations—



However, in the absence of an intermediate oxidation these reactions are not quantitative. 3. Oxalic acid in a solution containing 5 grms. of manganese sulphate and 12 c.c. of strong sulphuric acid per 100 c.c. is destroyed in a regular manner by chloric and bromic acid, with formation of hydrochloric or hydrobromic acid:—



Iodic acid acts as in the case mentioned in (2), only the yield is theoretical.

**New Method of Synthesis of Tertiary Alcohols by means of Organomagnesium Compounds.**—V. Grignard.—The author starts with a compound of the form  $\text{RC} \equiv \text{O} \text{MgX}$ , obtained as he has described in a former paper, and by performing on the second oxygen atom, a reaction identical with that on the first, he should obtain the complex  $\text{R}_1\text{R}_2\text{C} \begin{smallmatrix} \text{O} \\ \text{MgX} \end{smallmatrix}$ , the hydrolysis of which would give the ketone  $\text{R.CO.R'}$ . He finds, however, a scarcely appreciable quantity of the ketone formed, but instead a very good yield of the tertiary alcohol,  $\text{RC(OH):R'}$ .

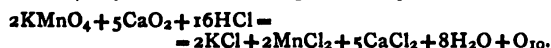
## MISCELLANEOUS.

**Royal Institution.**—On Tuesday next, February 23, Mr. E. Foxwell, late Professor of Economics and Finance at the Imperial University, Tokyo, will deliver the first of three lectures at the Royal Institution, at 5 o'clock, on "Japanese Life and Character"; and on Thursday (February 25), at the same hour, Prof. H. L. Callendar will commence a course of three lectures on "Electrical Methods of Measuring Temperature." The Friday Evening Discourse, on February 26, will be delivered by Mr. Alexander Siemens, his subject being "New Developments

in Electric Railways"; on March 4, by Prof. W. Stirling, on "Breathing in Living Things"; and on March 11, by Prof. F. T. Trouton, on the "Motion of Viscous Substances."

**Speed of the Reaction between Permanganate of Potassium and Oxalic Acid.**—R. Ehrmfeld.—In the absence of sulphuric acid the solutions of permanganate and of oxalic acid decomposed according to the equation  $8C_2H_2O_4 + 2MnO_4K = 2C_2O_4Mn + C_2O_4K_2 + 10CO_2 + 8H_2O$ . The reaction takes place rapidly at 50–60°. Towards the end of the reaction the liquor takes a reddish-yellow colour, and finally becomes cloudy; on boiling it forms brownish flakes on the surface. The author has examined the speed of the reaction at 20.3° by estimating at various times the amount of undecomposed permanganate by means of a solution of KI. The reaction between the permanganate and oxalic acid is a unimolecular reaction; in all cases the products formed by the reaction determine an increase in the speed of the reaction. This speed depends also on the concentration of the solutions, which shows that a portion of the undecomposed permanganate enters into the reaction.—*Zeit. Anorg. Chem.*, vol. xxxiii., p. 117.

**Iodometry of the Peroxides of Calcium, Strontium, Barium, Magnesium, and Sodium.**—E. Rupp.—The titration of the peroxides of the alkalis and the alkaline earths can be effected by the method described by the author for the estimation of the persulphates and the percarbonates. With all these oxides the general reaction is as follows:  $MO_2 + 4HCl + 2KI = MCl_2 + 2KCl + 2H_2O + I_2$ . The iodine set free is estimated with hyposulphite of soda. As for checking the results obtained by this method, this can easily be done by estimating the same oxides by  $KMnO_4$ ; by this means  $CaO_2$  gives the equation—



When used simultaneously, both methods constantly give concordant results.—*Arch. d. Pharm.*, vol. cxi., p. 437.

## MEETINGS FOR THE WEEK.

- MONDAY, 22nd.**—Society of Arts, 8. (Cantor Lectures). "Modern Book Printing," by Charles T. Jacobi.  
— Society of Chemical Industry, 8. "Duty-free Alcohol," by Thomas T. Tyrer.
- TUESDAY, 23rd.**—Royal Institution, 5. "Japanese Life and Character," by Ernest Foxwell, M.A.
- WEDNESDAY, 24th.**—Society of Arts, 8. "Mahogany and other Fancy Woods available for constructive and Decorative Purposes," by Frank Tiffany.
- THURSDAY, 25th.**—Royal Institution, 5. "Electrical Methods of Measuring Temperature," by Prof. H. L. Callendar, F.R.S.
- FRIDAY, 26th.**—Royal Institution, 9. "New Developments in Electric Railways," by Alexander Siemens, M.Inst.C.E.  
— Physical, 8. (At Royal College of Science, South Kensington). "A New Dilatometer," by Mr. Borniksen. "A Quartz-thread Vertical Force Magnetograph and Some Hints on the Preparation of Diagrams," by Dr. W. Watson. "On Structures in a Magneto-static Field," by G. W. Walker.
- SATURDAY, 27th.**—Royal Institution, 3. "The Life and Work of Stokes," by Right Hon. Lord Rayleigh, F.R.S.

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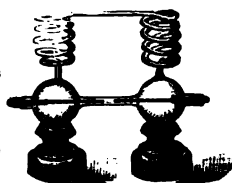
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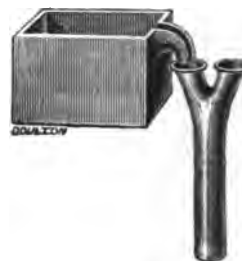
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## CONTENTS.

| ARTICLES:—                                                                                                      | PAGE |
|-----------------------------------------------------------------------------------------------------------------|------|
| Contributions to Our Knowledge of Radium, by W. Marckwald...                                                    | 97   |
| The Invariability of the Wave-lengths in the Spark and Arc Spectrum of Zinc, by J. M. Eder and E. Valenta ..... | 98   |
| On the Colorimetric Determination of Small Quantities of Phosphoric Acid and of Silica, by F. T. Veitch .....   | 101  |
| Precipitation and Separation by Weak Organic Bases, by E. T. Allen .....                                        | 103  |
| What Physical Chemistry has done for Chemistry, by H. C. Jones                                                  | 105  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                     | 106  |
| MISSINGS FOR THE WEEK .....                                                                                     | 107  |

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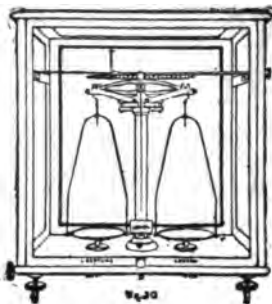
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# THE CHEMICAL NEWS.

Vol. LXXXIX., No. 2309.

## CONTRIBUTIONS TO OUR KNOWLEDGE OF RADIUM.

By W. MARCKWALD.

### I. ON THE SEPARATION OF RADIUM FROM BARIUM.

HITHERTO only one method has been available for the separation of a mixture of salts, richer in radium, from the radium-barium salts as obtained from the uranium minerals. By the fractional crystallisation of the chlorides (according to Curie) or, more advantageously, of the bromides (according to Giesel) it is possible to accumulate the radium salt to any desired extent in the more difficultly soluble fractions. This method of crystallisation is very detailed, because of the isomorphism of the barium and radium salts (cf. W. Marckwald, *CHEMICAL NEWS*, 1901, lxxiv., 190; Rinne, *Centralbl. f. Min. u. Geol.*, 1903, 134-141).

The idea that it might be possible to separate the two alkali earths from one another, by a suitable electrolytic method, induced me to arrange an experiment the result of which confirmed this conjecture. The concentrated aqueous solution of a quantity of radium-barium chloride\* was allowed to stand, and shaken up frequently with about a fifth of its weight of sodium amalgam. Only a very slight decomposition of water takes place, but a very considerable part of the sodium goes into solution as ion, while an equivalent quantity of radium and barium amalgam is formed. The proportion of radium to barium in the amalgam is by no means the same as in the solution, but is very much higher. If the amalgam is separated from the solution after the action has lasted one to two hours, decomposed with hydrochloric acid, and the small quantity of chloride resulting on the evaporation of the hydrochloric acid solution is compared on the phosphorescence screen with an equal quantity of the salt remaining in the original solution, then it appears, without any necessity for applying a more delicate method of measuring, that the first salt far exceeds the latter in activity.

By repeating the operation many times, after first properly neutralising the solution, we obtain from the amalgams fractions of salts, the activity of which gradually decreases.

In the above form, the new method of accumulating the radium offers no important practical advantages over the crystallisation method, for in both cases the aim in view is only reached by a process of fractionation. Whether it is possible to simplify the process for obtaining radium from the raw product by further development of this method does not yet appear. Meanwhile, this observation which I now communicate is of interest, because for the first time a difference is exhibited in the behaviour of radium and barium.

### II. ON THE PHOSPHORESCENCE OF THE ANHYDROUS RADIUM BARIUM CHLORIDE.

It is well known that mixtures of anhydrous radium and barium chloride, even when they contain very little active salt, are strongly phosphorescent, while the hydrous crystalline salts either do not phosphoresce at all or only very slightly. This phenomenon is to be ascribed to the fact that phosphorescence is excited in anhydrous barium chloride by the Becquerel rays, both by  $\alpha$ - and  $\beta$ -rays, and

not in the hydrous crystalline salt. Thus, if a little rod coated with radio-tellurium, which only emits  $\alpha$ -rays, is brought up to the first salt, it becomes brightly luminous, as well as at the approach of a radium preparation enclosed in an aluminium capsule, which gives out only  $\beta$ -rays. On the other hand, the crystalline hydrous barium chloride shows no phosphorescence under the same conditions.

### III. ON INDUCED RADIO-ACTIVITY.

The interesting publication of F. v. Lerch (*Ann. d. Phys.*, [4], xii., 745) on the induced thorium activity leads me to communicate some observations on the induction of the radio-activity of radium solutions on metals, although the research is not yet completed.

Freshly prepared solutions of radium salts, which had been kept in a dry state for a long time, and had thus reached their maximum activity, are, according to Curie's and Giesel's researches, strongly active, but the activity rapidly dies away—according to Rutherford because the emanation is given up—until it has reached a minimum. If in the solution of the radium barium chloride mixture, as it is obtained directly from the Joachimsthal pitch-blende, metals are immersed after the solution has stood for many days, these are rendered only very feebly active by induction, but if freshly prepared solutions are used, after remaining a short time in the solution certain metals become very strongly active. This activity reaches its maximum in from 15 to 30 minutes, and slowly diminishes in the course of a day after removal from the solution. The induced metal emits both  $\alpha$ - and  $\beta$ -rays, which in the case of some metals are strong enough to enable the phosphorescence of the zinc blende screen by direct radiation ( $\alpha$ -radiation), as well as that of the barium platinum cyanide screen, through a layer of cardboard ( $\beta$ -radiation) to be seen even in an imperfectly darkened room. A noteworthy difference is shown between the different metals as regards the intensity of the induction. Under exactly the same conditions, the degree of induced activity appears to depend generally on the position the metals occupy when arranged in a series, according to their contact forces. At any rate the induction decreases in the metals magnesium, zinc, tin, copper, silver, bismuth, palladium, in this order, and the differences are so great that in observing the action on the phosphorescence screen it is at the most only in the case of neighbouring members of the series that there can be any doubt.

If many strips of the same metal are immersed in the same solution of the salt one after another at short intervals, i.e., after about one to two hours, generally no noticeable diminution of the inductive action is to be observed. An exception must be made only in the case of magnesium. While the first strip becomes very strongly active, all the subsequent ones are only feebly acted upon. This phenomenon must be ascribed to the fact that the solution, by the immersion of the first magnesium strip, becomes alkaline, if only to a very slight extent. This is inferred chiefly from the fact that magnesium is made very strongly active, and the action is continuous, if the solution is kept acid by the addition of a small quantity of hydrochloric acid.

It might be thought that in acid solution the surface of the metal is kept clean, while in neutral solution it is covered with a layer of oxide, and that this hinders induction. Hence a little ammonium chloride was added to the neutral solution of the radium salt; thus the magnesium oxide was dissolved, the solution becoming alkaline. In this case the strong inductive action was absent.

These observations led me to examine the behaviour of zinc and copper in a solution of radium chloride made feebly alkaline by ammonia, and in another made feebly acid by hydrochloric acid. It then appeared that the zinc, which is chemically allied to magnesium, behaved in an exactly analogous manner, while the copper was about equally strongly acted upon in both solutions.

In conclusion, it must be mentioned that all these experi-

\* For this experiment a mixture of salts was used, which had been obtained from Joachimsthal pitch-blende without further increasing the proportion of radium.

ments were confined to the feebly active radium preparation mentioned above. Whether radium salts containing high percentages will give analogous phenomena has not yet been examined.—*Berichte der Deutsch. Chem. Gesell.*

### THE INVARIABILITY OF THE WAVE-LENGTHS IN THE SPARK AND ARC SPECTRUM OF ZINC.

By J. M. EDER and E. VALENTA.

THE breadth of the lines in the arc and spark spectrum is a complicated, and as yet only partially known function of the temperature, pressure, and the density of the luminous layer of vapour (H. Kayser\*), and is probably dependent on the kind of electric excitation, and of the dielectric (Hartmann†). The experimental observations of these phenomena of broadening are as yet by no means finally concluded. In particular it is not yet known whether the intensity maximum—in a certain measure the centre of gravity of a spectral line—is displaced when it broadens or not, which is a question of fundamental importance for the whole of spectrum analysis.

Actual displacements of spectral lines (*i.e.*, clearly demonstrable alterations of the wave-lengths) have undoubtedly been proved by Humphrey and Mohler‡ as a consequence of variable external pressure.

But F. Exner and E. Haschek went distinctly further when they said that these displacements occurred with greater intensity in the spark spectrum than in the arc, and depended not only upon the external atmospheric pressure but also on the partial density of the vapour under examination (Exner and Haschek, "Wellenlängen Tabellen für Spektralanalytische Untersuchungen," Leipzig und Wien, 1902, p. 13). They further stated that many spark spectra show considerable displacements of the spark lines as opposed to the analogous lines in the arc spectrum (in both cases at ordinary external atmospheric pressure), and tried to explain this by the pressure phenomena in the spark (These *Sitzungsber.*, 1897, *et seq.*), and would calculate from the magnitude of the displacements of the lines the magnitude of the pressure in the spark; E. Haschek and H. Mache (These *Sitzungsber.*, 1898) tried to demonstrate the pressure in the spark directly, but could obtain no agreement of the values calculated from the "displacement of the lines" with the observed values. From his further researches, E. Haschek (These *Sitzungsber.*, Bd. cx., Abt. II.a, März, 1901, p. 181) afterwards concluded that the laws which hold good for the displacements of the lines in the spark are different from those which obtain in the case of the arc, and that therefore a calculation of the spark pressure from the displacement of the lines is not admissible; the spark pressure observed from Haschek's measurement showed so little agreement with that calculated by him that he himself said:—"The calculated pressures differ so much among themselves that there can be no doubt that other causes are responsible for the displacements of the lines" (Haschek, *loc. cit.*). As such another cause of the "line displacements" Haschek mentions "the increasing density of the luminous vapour"; with equal increase of the vapour density it is said that "the lines in the spark spectrum are displaced more than those in the arc spectrum"; thus, for

example, according to Haschek, in the zinc spectrum the lines  $\lambda_{3282}$ , 3302, 4680, 4722 have in the spark spectrum a wave-length greater by 0.03 to 0.16 Angström units than in the arc; in other cases Haschek mentions displacements up to 0.2 A.U. for elements with many lines, and up to 0.07 A.U. for elements with few lines, which are very considerable amounts, lying far beyond the limits of errors of observation permissible with grating spectra. As a proof of the correctness of these observations he performed numerous wave-length measurements (with different elements, *e.g.*, B, Al, Zn, Ca, Si, Zr, Cr, Ti, Ce, Va, &c.\*). From these statements it seemed as if the existence of real line displacements was certainly proved for the spark (as opposed to the arc lines) for ordinary external atmospheric pressure. Also the quantity of the vaporising substance, according to Haschek, is said to have an appreciable influence on the actual wave-length of the lines, so that in his opinion a method of quantitative spectrum analysis could be based on the measurement of these so-called line displacements (These *Sitzungsber.*, Bd. cxi., Abt. II.a, February, 1902, p. 232). For example, for pure zinc (spark spectrum) the wave-length 4722.510 is said to be correct, while it amounts only to 4722.399 for the same zinc line, using a 5 per cent zinc alloy instead of pure zinc as electrode. Thus the partial density in the luminous vapour would have a definite influence on the wave-length.

The theory relating to the dependence of the wave-length of the spectral lines on the partial density of the vapour under examination occupies a prominent position in the work by F. Exner and G. Haschek, "Wellenlängentabellen für Spektralanalytische Untersuchungen auf Grund der Ultra-violetten Funkenspektren der Elemente" (Leipzig und Wien, 1902, I. Teil., p. 13). The comprehensive numerical results which the above authors give as obtained in their wave-length measurements would force us to acknowledge the value of this evidence in their favour, if we could suppress our well-grounded doubt concerning the value of the evidence afforded by their photographic material.

On an earlier occasion we had already obtained results which did not agree with their statements in the case of the spark spectra of calcium and lithium, as we pointed out at the time (we drew attention to the fact that photographic errors might have occurred), and when we began new wave-length measurements in the zinc spectrum we again met with experimental results which were contradictory to Haschek's statements. Also our results with regard to the spectra of alloys both poorer and richer in zinc do not agree with Haschek's statements and measurements.

H. Kayser, of Bonn ("Handbuch der Spektroskopie," Bd. ii., pp. 297, 308, 309, and 310), also opposed Exner and Haschek's statements; he stated that during his spectroscopic researches on the electric arc he had never observed any influence exerted by the mass of the element present on the wave-length, and according to his view the partial pressure of the vapour in the arc could produce no displacements, as can be easily proved if we work with apparatus of considerable resolving force, while otherwise apparent displacements may result. As regards the comparison of the wave-length in the spark with the arc lines, and the influence of the partial pressure on displacements of the lines in the spark spectrum, at present no further experimental confirmation exists regarding Exner and Haschek's assumption of displacements of the lines.

It may here be stated explicitly that it is not a question of the breadth of the lines in the so-called asymmetrical phenomenon of broadening, which has been known for many years, and which amongst other things manifests itself in

\* "Handbuch der Spektroskopie," Leipzig, 1902, Bd. II., p. 296.

† *Sitzungsber. der Königl. Preussischen Akad. der Wiss.*, Berlin, 1903, p. 40, and 234.

‡ *Astrophys. Journ.*, 1896 and 1897. Humphrey and Mohler increased the pressure of the gas, in which the arc is produced, to several atmospheres, and observed the displacement of some spectral lines towards the red, and they found that the increase of wave-length is proportional to the pressure of the surrounding gas.

§ No notice at all is taken in this treatise of displacements of lines caused by rapid movement of the sources of light in the radius of vision in the sense of Doppler's principle, which displacements are used for the measurement of the movement of the stars in the radius of vision.

\* Meanwhile, N. A. Kent has proved that in the case of the titanium lines Haschek's displacements of 0.13 A.U. do not exist (*Astrophys. Journ.*, 1903, Bd. xvii., p. 286).

† Eder and Valenta, "Über das Funkenspektrum des Calciums und Lithiums und Seine Verbreiterungs und Umkehrungserscheinungen," *Denkschr. Akad. der Wiss. Wien.*, 1898, Bd. lxxvii.

such a way that with pure metal electrodes (e.g., zinc, lead, &c.) spectral lines in the spark from a jar are decidedly broadened towards the red, and, on the other hand, the same lines become fairly well defined if these metals in the electrodes occur only as small impurities (thus with small partial density) in the vapour. In the case of Exner and Haschek's phenomenon of the displacement of the lines, it is a question rather of the exact wave-length of the intensity maximum of the lines\* which, in a certain measure, represents the "centre of gravity" of the lines concerned.

If we take as basis for such measurements the intensity maximum of the lines in question, which is now recognised both generally and also by Haschek (*These Sitzungsber.*, 1902, Bd. iii., Abt. II., p. 232) as the only permissible method of taking such measurements with correct photographic procedure, and the most accurate possible resolution of the spectra, we arrive at results which contradict Haschek's statements.

We first attacked the zinc spectrum and devoted ourselves exclusively for a long time to eight zinc lines, which could be taken as typical, and which, in addition to other similar spectra, were brought forward by Haschek as affording specially valuable evidence in favour of his theory.

Our grating is an excellent large Rowland's concave grating with a radius of curvature of 15 English feet (i.e., the same radius of curvature as that of the grating used by Exner and Haschek), which H. Kayser, of Bonn, who saw our spectrum photographs, calls a grating "of the very first order." Now, with asymmetrically broadened lines—and Haschek's research only applies to such—the possibility of finding the intensity maximum with certainty depends on restricting the broadened extended part by the most careful photographic procedure. We worked with a slit of 0.008 to 0.03 m.m. breadth (sharp steel knife edges), which last even gave good definition of the lines, ascertained the true photographic focus of the grating by empirically removing† the photographic plates‡ m.m. at a time, and bent the photographic mirror plates confocally with the curvature of the image. We arranged the photographing of the lines one over the other in such a way that a blind for the slit, which could be easily removed to and fro, could be interposed before the photographic apparatus, and by moving this blind every line could be photographed in three parts one after the other. At each separate operation the whole grating was fully illuminated. The illumination of the plate was short.

No agreement has yet been arrived at regarding the idea of a "short illumination" or "minimum illumination" with the best clearness and sharpness and correct development. As long as this is the case, differences of observations with regard to the so-called displacement phenomena of spectral lines, as E. Haschek repeatedly describes them, in contradiction to other spectroscopists, will exist, and it will be difficult to avoid deception. In order to fix definitely the sharpness of the lines, the breadth of the measured line should not only be expressed in Angström units (A.U.), but the "degree of blackening" of the line in question on the photographic negative should also be given; the latter should be fairly small. It may be regarded as a criterion whether a photograph will meet the strictest requirements if an asymmetrically broadened line is obtained with medium or least blackening with a certain minimum breadth. We measured the breadth of the sharp iron lines resulting under favourable experimental conditions in the arc spectrum; they were, for example, 0.03 to 0.04 m.m. broad with a slit of 0.03 m.m. breadth (with minimum illumination) when they produced, measured in Hartmann's microphotometer a blackening of 0.5 to 1.0; while the zinc

lines, when strongly subjected to the broadening under exactly the same conditions, were never narrower than 0.06 m.m. in the negative of the spectrum. If the scale is reduced to A.U. the zinc line  $\lambda 4810$  has thus in the arc a breadth of 0.07 A.U. against 0.19 A.U. in the spark (in both cases with minimum illumination); nevertheless, we can easily prove the coincidence of the two lines at the position of their greatest light intensity. But if we illuminate the zinc spark twice as long, the asymmetrical broadening is so marked that the total breadth of the line amounts to 0.53 to 0.71 A.U., and then it is difficult to find the maximum of the light intensity; the central part of the line is shifted towards the red; but it is still possible to think that the maximum has been reached, and thus an apparent displacement of 0.008 A.U. may be found, which increases to 0.02 A.U., and with long illumination, bad development, and imperfect reading in consequence of feeble light, can amount to still more. The reason for this apparent displacement is to be found in the fact that the central part of the line is not more resolved. Also the placing has an appreciable influence. When it is not perfectly exact, asymmetrically broadened lines become unrecognisable and disappear.

It is difficult to decide whether this is sufficient to explain the very contradictory statements of different spectroscopists who have photographed one and the same phenomenon, but it is a fact that by carefully carrying out the spectrographic process as described, we succeeded in proving the non-existence of all alleged displacement phenomena, which Haschek described for zinc, as well as for other spectra.

The arrangement of our experiments, as regards the production of the spark discharges in question between zinc electrodes, exactly agreed with those experimental conditions under which Haschek thought he had proved the existence of "line displacements." In his "*Spektralanalytische Studien*" (*These Sitzungsber.*, 1901, Bd. cx., Abt. II., p. 200) he says expressly that the lines were displaced more with the Ruhmkorff inductor and hammer interruption, than when a similar inductor with Wehnelt interruption, or when a transformer was used. Hence, we also used powerful Ruhmkorff induction coils with hammer interruptors; we had two large inductors, striking at a distance of 12 to 25 c.m., with correspondingly large condensers, so that a real displacement phenomenon could not possibly have escaped us.

But under these conditions the asymmetrical broadening phenomena of the zinc lines  $\lambda 4680$ ,  $\lambda 4722$ ,  $\lambda 4810$ , &c., appeared clearly, and in our experiments we obtained at once one of the phenomena which Exner and Haschek call displacement phenomena. Fig. 1. of our heliographic table shows two zinc spectra\* accurately photographed one above the other, which immediately make it clear how the "displacement phenomenon" appears. As a matter of fact, from a superficial inspection one would be inclined to pronounce in favour of a displacement of the lines, although a displacement of the true intensity maximum does not actually occur. Similar false "displacement phenomena" also appear under other different experimental conditions; if the photographs of the zinc lines are illuminated too long they present this appearance, if we produce them on the one hand with pure zinc electrodes, and on the other with zinc alloys, which only contain a little zinc, which is interpreted by Exner and Haschek as the influence of the partial pressure of the vapour; also with defective definition of the spectra, the zinc lines taken for the sake of comparison in the arc (narrow lines) and in the spark (asymmetrically broadened) generally look similar. But it may at once be stated that the two spectra in Fig. 1. were obtained from one and the same zinc spark, with short and long exposure (one-half minute to five minutes),† and that the supposed displacement of lines is only the photographic expression of the asymmetrical broadening, as the photograph of a wedge-shaped, outlined

\* Specially discussed and illustrated by us in our treatise "*Funkenspektrum des Calciums und Lithiums und Seine Verbreitungen und Umkehrungserscheinungen*" (*Wiener Akad. Denkschr.* 1898).

† In such spectrum photographs, in which the highest definition is required, a change of 1 m.m. in the plane of position (with a total focus of 6 m.) produces very considerable disturbances.

\* Facsimile of the original negative in heliogravure.

† N.B.—The finer lines in Spectrum 2 are iron lines photographed in.



streak of light would show it with lengthened exposure, even if the intensity maxima had remained quite constant. The spectra, when very shortly illuminated, give only the intensity maximum of the lines which coincide, while with longer illumination the line of the spark spectrum broadens more towards the red than towards the side of shorter wave-length, and with rather longer development of the photographic plate the intensity maximum of the line is united with the asymmetrical broadening. After the conditions under which the phenomenon in question appears are explained, we can pass to the description of the results of our own experiments in which those circumstances liable to give rise to error were carefully avoided. When every precaution is taken to ensure minimum illumination, the plates are correctly developed and the spectra best defined, it appears:—

1. That at ordinary atmospheric pressure there occur no displacements, which are said to appear, according to Exner and Haschek, in the spark spectrum as opposed to the arc spectrum.

2. That also in the spark spectrum no displacements of the lines exist, which were to be ascribed to small quantities of the element present in the vapour, *i.e.*, that any dependence on the partial pressure in the sense of Exner and Haschek's assumptions cannot be proved.

In both cases only the known variable asymmetrical broadenings of the lines occur, but the brightness maxima of the lines coincided perfectly in our experiments.

We studied the same zinc lines in which Haschek observed displacements in the spark spectrum, and first determined their wave-length as accurately as was practicable, using Rowland's standard, corrected by Kayser ("Normale aus dem Bogenspektrum des Eisens," *Ann. der Physik. und Chemie.*, 1900, 4 Folge, Bd. iii., p. 195); the lines were measured under the microscope, each line at least ten times on different spectrum photographs, so that our wave-length observations are correct to 0.002 Å.U.

As according to Haschek the lines in the arc spectrum of zinc show the normal wave-length, and with small partial pressure, in Haschek's sense, are better for ascertaining the normal wave-length (without any fear of the disturbing so-called "displacement phenomena"), we chose for the formation of the zinc arc spectrum, brass wire (in the small electric arc),\* which according to chemical analysis contained 36.9 per cent zinc and 63.1 per cent copper.

The carefully determined wave-lengths of the zinc lines of such an arc light between brass electrodes gave for the arc spectrum:—

|                 |       |            |
|-----------------|-------|------------|
| Zinc—III. order | .. .. | λ 3302.715 |
| " " "           | .. .. | 3303.068   |
| " " "           | .. .. | 3345.141   |
| " " "           | .. .. | 3345.698   |
| " " "           | .. .. | 3282.451   |
| " II. "         | .. .. | 4680.327   |
| " " "           | .. .. | 4722.333   |
| " " "           | .. .. | 4810.719   |

In the same field of vision in the arc spectrum, next to them came the copper lines of the third order, λ 3247.671 and 3274.090 (Rowland) which in the arc appear reversed (symmetrically broadened), while the arc spectrum of iron, photographed as normal, contains a trace of copper and gave a fine black copper line, which coincided perfectly with the phenomenon of inversion.†

Our heliographic table, Fig. II., 1 and 2, shows the exact spectrum photograph of the arc spectrum of a zinc-copper alloy; it is clear how with increasing illumination all the lines of the arc spectrum remain fairly sharp, and broaden symmetrically. Pure zinc gives in the spark

spectrum λ 4680, 4722, 4810, with increasing illumination asymmetrically broadened strongly towards the red, but with exact minimum illumination, the brightest part of the line appears sharp enough in the strongest spark from a jar to fix exactly its wave length, and to be able to establish under the microscope its coincidence with the arc line, which is evident from our table (Fig. II., 1—4). With the zinc lines λ 3282, 3302, 3345.1, we found likewise perfect coincidence in the arc lines and spark lines; the zinc spark lines λ 3345.1 and 3302.7 are easily reversed, and then coincide perfectly with the (unreversed) arc line. With over-illumination a broadening of this line towards the ultra-violet occurs. The zinc line 3345.698, which is to be obtained well-defined and clear, both in spark and arc, without difficulty, and 3303.068 are considered by Haschek himself as not capable of displacement, because their tendency to remain well-defined even with inexact times of illumination prevents any error which could exist with defective definition in the case of the zinc lines 4680, 4722, &c., which broaden asymmetrically.

Fig. III. shows in Spectrum 1 and 3 the spark spectrum of pure zinc; Spectrum 2 in the middle is the arc spectrum of the zinc-copper alloy, and over all three is extended the arc spectrum of an iron wire containing traces of copper. We see the perfect coincidence of the reversed copper arc line λ 3247, and 3274 with the unreversed copper spark line. The zinc lines, λ 4722 and 4810 (Fig. III.), are rather much illuminated and developed to strong blackness, by which means the arc lines of the middle spectrum have broadened on both sides fairly uniformly, while the spark lines show asymmetrical broadening and the beginning of apparent displacements, which disappears with minimum illumination and shorter development, after which the perfect coincidence analogous to Fig. II. becomes undoubtedly evident.

In the zinc lines, λ 3345 and 3302 of Fig. III., we further see that these zinc lines in the spark, as compared with the arc lines, are by no means displaced towards the side of greater wave-length, as Haschek (*loc. cit.*) erroneously states. We see this still more clearly in the measurement of the original negative under the microscope of the measuring apparatus.

The supposition that Haschek had badly-defined spectra gains in probability owing to the fact that the finer structure of some zinc spark lines, which is very remarkable, escaped him. While the asymmetrical very marked broadening of the zinc lines 4680, 4722, 4810 towards the red did not escape Haschek, he did not notice that the zinc lines λ 3345.1 and 3302.7 show a tendency to slight broadening towards the side of shorter wave-length, and hence they lose the tendency to apparent "displacements" towards the red. Nevertheless, Haschek finds the lines in the spark specially strongly displaced towards the red (as compared with the arc), and we find, on the contrary, that here with correct short exposure generally no displacement, but coincidence, results.

After we had failed to establish the results quoted as relating to the line displacements in the spark as opposed to the same lines in the arc, there still remained further experiments to be arranged on the influence of the partial pressure of the luminous zinc vapour on the displacement of the spectral lines. For this purpose we subjected to an accurate investigation the spark spectrum of pure zinc and that of alloys of 1 per cent zinc + 99 per cent lead, as well as 50 per cent zinc + 50 per cent lead and 36.9 per cent zinc and 63.1 per cent copper. In the case of alloys poor in zinc, according to the experience of all spectroscopists, which was first published long ago and is now generally known and never disputed—and this holds good for all spectra, especially with asymmetrically broadened lines—all those lines, even with increasing illumination, remain fairly sharp, which, in alloys rich in zinc, undoubtedly show in a high degree the tendency to asymmetrical broadening. Thus far the partial pressure undoubtedly exerts an influence on the breadth of the spectral line.

\* The brass electrodes were used directly for the production of the arc.

† In agreement with these experiments of ours, Exner and Haschek in their "Wellenlängentabellen," Bd. 1, p. 21, speak of the two copper lines 3247 and 3274 as not capable of being displaced.

Thus, with increasing time of illumination, the asymmetrical broadening phenomena in the case of alloys rich and poor in zinc are not the same; thus, if we measure on the more strongly illuminated plates the wave-length of alloys rich and poor in zinc by placing on the middle of the badly-defined lines, apparent line displacements appear with rising partial pressure (analogous to the photograph in Fig. 1.), which, however, we at once recognise as apparent displacements if we have returned to minimum times of illumination and have obtained the best defined spectra.

A 1 per cent zinc alloy (1 per cent zinc + 99 per cent lead) naturally gives the separate zinc lines in the spark less clearly than one richer in zinc, e.g., a 50 per cent zinc alloy. A preliminary test for our strongest spark (Ruhmkorff) showed that the poorer alloy (1 per cent) must be illuminated for about fifteen minutes, as compared with about thirty seconds for the richer (50 per cent) in order to get the zinc lines 4680, 4722, 4810 approximately equally sharp (equally bright) on the photographic plate for both alloys. Hence in our preliminary experiment in the first case the zinc lines would be thirty times brighter than in the second.

If, now, we use equivalent times of illumination and approach the minimum time of illumination, which in both cases gives faint, just measurable lines in the spectrogram, in the spark spectrum the wave-lengths of the zinc lines 4680·327, 4722·333, and 4810·719 in 1 per cent, 4 per cent, and 50 per cent alloys, and in pure zinc, are exactly the same, and we could never—even with the strongest jar spark—see Exner and Mache or Haschek's "line displacements."

Haschek (These *Sitzungsber.*, Feb., 1902, Bd. cxi., Abt. II.a, pp. 238 and 240) states that alloys with a little zinc (4 per cent zinc) give in the spark spectrum zinc lines of wave-lengths  $\lambda$  4722·399, but with 50 per cent zinc this becomes 4722·434, which would be a considerable displacement towards the red with increasing partial pressure; he finds the zinc line 4680 in alloys poor in zinc (4 per cent zinc) too faint to be measured. But this line 4680, even in 1 per cent alloys, can be measured (this is shown clearly in our table, Fig. IV., 4) if we choose the time of illumination properly. Haschek has obviously under-exposed the alloy poor in zinc and over-exposed the richer alloy, and was deceived by an asymmetrical broadening of the lines with defective definition of his spectra. As far as we can see, Haschek applies his theory of displacement only to asymmetrical broadened lines; with the sharp lines not even the apparent displacement of the lines is to be seen in the spark spectrum as opposed to the arc spectrum.

Thus our results are:—

1. In the spark spectrum of zinc, as opposed to the arc spectrum, no displacements of measurable magnitude appear.

2. The mass of the element present, or the partial pressure of its vapour, produces no displacement of the lines of the spark spectrum, and hence Haschek's system of quantitative spectral analysis falls to the ground; on the contrary, those conclusions which presuppose the constancy of the spectral lines (displacements of the spectral lines in the radius of vision according to Doppler's principle, &c.), and which appeared doubtful owing to Haschek's deductions, are further confirmed by our observations.—*Sitzungsber. der Kaiserl. Akad. der Wiss. in Wien.*, Bd. cxii., Abt. II.a, October, 1903.

Formation and Saccharification of Retrograde Amidon.—L. Maquenne.—The author concludes that amylocellulose is not a single principle, but a mixture of several different products of condensation which have the common characteristic of not being coloured by iodine. It has also the distinctive characteristic of offering a variable resistance to the solvent action of amylase.—*Comptes Rendus*, cxviii., No. 4.

# ON THE COLORIMETRIC DETERMINATION OF SMALL QUANTITIES OF PHOSPHORIC ACID AND OF SILICA.\*

By F. P. VEITCH.

(Concluded from p. 91).

## The Colorimetric Determination of Silica.

In their study of the effect of silica on the development of colour, Woodman and Cayvan (*Journ. Am. Chem. Soc.*, xxiii., 96) found that while some colour was developed instantly, it did not reach the maximum intensity in less than from one and one-half to two and one-half hours, and their results further show that the colour due to the formation of the ammonium silico-molybdate is about one-tenth that produced by an equivalent amount of ammonium phosphomolybdate. This reaction apparently was used first by Jolles and Neurath (*Zeit. Angew. Chem.*, 1898, p. 315) for the determination of silica in waters. For our determination of silica a solution of sodium silicate was prepared and the silica determined gravimetrically. Portions of this solution were compared with the standard phosphate solution under identical conditions as to temperature, nitric acid, molybdate solution, and volume. The data obtained are given in the following table:—

TABLE VII.—Readings of SiO<sub>2</sub> in Terms of P<sub>2</sub>O<sub>5</sub>.

| SiO <sub>2</sub> in solution read.<br>M.grm. | P <sub>2</sub> O <sub>5</sub> required to equal colour produced by SiO <sub>2</sub> .<br>M.grms. | 1 m.grm. SiO <sub>2</sub> equals P <sub>2</sub> O <sub>5</sub> .<br>M.grms. |
|----------------------------------------------|--------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------|
| <i>Preliminary Series.</i>                   |                                                                                                  |                                                                             |
| 0·10                                         | 0·19                                                                                             | 1·90                                                                        |
| 0·10                                         | 0·175                                                                                            | 1·75                                                                        |
| 0·20                                         | 0·35                                                                                             | 1·75                                                                        |
| 0·20                                         | 0·37                                                                                             | 1·85                                                                        |
| 0·30                                         | 0·51                                                                                             | 1·70                                                                        |
| 0·30                                         | 0·57                                                                                             | 1·90                                                                        |
| 0·50                                         | 0·92                                                                                             | 1·84                                                                        |
| 0·51                                         | 0·90                                                                                             | 1·78                                                                        |
| 0·70                                         | 1·35                                                                                             | 1·93                                                                        |
| 0·70                                         | 1·28                                                                                             | 1·83                                                                        |
| 1·00                                         | 1·80                                                                                             | 1·80                                                                        |
| Average .. .. .                              |                                                                                                  | 1·82                                                                        |
| <i>Final Series.</i>                         |                                                                                                  |                                                                             |
| 0·068                                        | 0·1265                                                                                           | 1·86                                                                        |
| 0·136                                        | 0·2464                                                                                           | 1·81                                                                        |
| 0·204                                        | 0·3741                                                                                           | 1·83                                                                        |
| 0·272                                        | 0·4785                                                                                           | 1·78                                                                        |
| 0·340                                        | 0·5907                                                                                           | 1·73                                                                        |
| Average .. .. .                              |                                                                                                  | 1·80                                                                        |

The table shows that the intensity of colour produced by the silica is on an average 1·8 times that produced by an equivalent amount of phosphoric acid. That is, the phosphate readings divided by 1·8 or multiplied by 0·55 equals SiO<sub>2</sub>.† It will be seen from the final results, in which most reliance is placed—as they were obtained after considerable experience with the method—that there is a gradual decrease in this factor with increasing quantities of silica. The time required for the full development of the colour was much shorter than found by Woodman and Cayvan, apparently reaching the maximum in less than twenty minutes, after which time it remained stationary for some time, and then slowly faded.

On the basis of this work the procedure which I have adopted for the determination of silica and phosphoric acid in soil solutions is as follows:—

The water or extract is tested for iron by adding potassium ferrocyanide to the acidified solution. The absence

\* From the *Journal of the American Chemical Society*, xxv., No. 2.

† These results differ greatly from those given by Woodman and Cayvan, but so far no time has been available for further study of this point.

of interfering amounts of iron having been shown, a measured volume of the water or soil extract is freed from suspended matter by filtration or by passing through a Chamberland filter (reject the first 100 c.c. that passes), or by evaporating to dryness and filtration, or in some cases where the water is but slightly turbid the turbidity or colour is corrected for by determining its amount in terms of the standard, the reading thus obtained being afterwards subtracted from the final readings. Add to the clear extract 5 c.c. of nitric acid (sp. gr. 1.07) and 4 c.c. of molybdate solution. Place in the camera, allow ten to thirty minutes for development of colour, and compare with a standard phosphate solution, which may conveniently contain 10 parts per million of phosphorus pentoxide and be contained in a sliding tube connected by rubber tubing with a side neck tube graduated in cubic centimetres within the camera. (The colour of the standard is not affected by the rubber tube during one working day, but the standard should be made fresh each day). The readings thus obtained (several should be made and the average taken) minus the reading for turbidity, when calculated to a volume of 100 c.c., equals  $P_2O_5 + SiO_2$  in parts per million of solution.

Another measured portion of the water or extract is evaporated to dryness twice, with a filtration between the evaporations in a porcelain or platinum dish with 3 c.c. nitric acid (sp. gr. 1.07) plus a little magnesium nitrate,\* heated two hours in a water-oven, 5 c.c. nitric acid (sp. gr. 1.07) added, filtered, washed to about 45 c.c., placed in a camera, and compared. If coloured, the reading is noted, and is finally subtracted from the total reading. Add 4 c.c. of ammonium molybdate, and thoroughly mix. Place in the camera, and compare after two to five minutes. The corrected reading calculated to volume of 100 c.c. is  $P_2O_5$  in parts per million of solution. This reading subtracted from the  $SiO_2 + P_2O_5$  reading and the difference multiplied by 0.55 gives the silica.

Where the original solution is too much coloured with organic matter to be accurately corrected for by reading the colour thus produced against the standard phosphate solution, it is necessary to evaporate with about 0.1 gm. magnesium nitrate\* and burn off the organic matter, take up with water + 3 nitric acid, evaporate to dryness, and heat two hours in the water-oven. Add 5 c.c. nitric acid and proceed as above. Readings =  $P_2O_5$ . In this case silica is not determined.†

In pure solutions the method gives very close readings and very accurate results on dilute solutions. The average error has been well within 0.2 part per million of the solution read where these solutions did not contain more than 10 parts per million and the readings fall below 60 scale divisions on the colorimeter tube. This insignificant error of reading may give rise to larger differences when calculated on the basis of the dry soil. Should the solution to be examined contain more than 10 parts per million, its dilution to this strength and subsequent calculation to the basis of the original solution or to the dry soil may so multiply this error of reading as to render the results valueless. This has been a serious handicap to the method. The use of more concentrated solutions is obviously the rational way out of the difficulty. It is also obvious that here we are limited to the use of concentrations from which the phosphomolybdate will not precipitate, within an hour or so. As solutions of greater concentration than 10 parts per million cannot be read closely in 25 m.m. tubes, with ordinary light, and as larger tubes were not available, I have not tried greater concentrations in larger tubes, but in several experiments using a strong standard (between 50 and 100 parts per million), in shallow depths in the 25 m.m. tubes, it was not found possible to read closer than

The results given in the following table have been obtained in the course of the work:—

TABLE VIII.—Determination of  $SiO_2 + P_2O_5$  in Miscellaneous Samples.

|                                                                | Parts per million. |                |                   |                 |
|----------------------------------------------------------------|--------------------|----------------|-------------------|-----------------|
|                                                                | $SiO_2$ present.   | $SiO_2$ found. | $P_2O_5$ present. | $P_2O_5$ found. |
| Sodium phosphate solution, No. 1 .. .. .                       | —                  | —              | 49.6              | 48.5            |
| Sodium phosphate solution, No. 2 .. .. .                       | —                  | —              | 99.1              | 101.4           |
| Sodium silicate solution ..                                    | 182.0              | 180.5          | —                 | —               |
| Soil extract—No. 13.. ..                                       | —                  | 24.4           | —                 | 2.0             |
| " 14.. ..                                                      | —                  | 23.4           | —                 | 2.95            |
| " 15.. ..                                                      | —                  | 21.2           | —                 | 2.95            |
| " 16.. ..                                                      | —                  | 19.1           | —                 | 2.95            |
| 1st foot.. ..                                                  | —                  | 17.9           | —                 | 7.3             |
| 2nd " .. ..                                                    | —                  | 8.2            | —                 | 1.0             |
| 3rd " .. ..                                                    | —                  | 7.8            | —                 | 3.1             |
| 4th " .. ..                                                    | —                  | 11.9           | —                 | 1.4             |
| Soil No. 2806, sandy truck soil .. .. .                        | —                  | 2.0            | —                 | 10.0            |
| Soil No. 2795, loam soil ..                                    | —                  | 38.0           | —                 | 8.0             |
| Soil No. 3, clay soil .. ..                                    | —                  | 30.0           | —                 | 8.0             |
| Soil No. 2804 clay soil ..                                     | —                  | 28.0           | 3 grav.           | 4.0             |
| Sodium phosphate solution—                                     |                    |                |                   |                 |
| Original solution (readings made for Prof. F. K. King) .. .. . |                    |                |                   | 73.1            |
| First 50 c.c. through Chamberland filter.. ..                  |                    |                |                   | 59.1            |
| Second 50 c.c. " .. ..                                         |                    |                |                   | 67.6            |
| Third 50 c.c. " .. ..                                          |                    |                |                   | 71.8            |
| Left in 50 c.c. in " .. ..                                     |                    |                |                   | 80.3            |
| Rinsings of " .. ..                                            |                    |                |                   | 80.3            |
| Original solution .. .. .                                      |                    |                |                   | 80.25           |
| First 50 c.c. through Chamberland filter.. ..                  |                    |                |                   | 54.9            |
| Second 50 c.c. " .. ..                                         |                    |                |                   | 71.8            |
| Third 50 c.c. " .. ..                                          |                    |                |                   | 84.5            |
| Left in 50 c.c. in " .. ..                                     |                    |                |                   | 97.2            |
| Rinsings of .. .. .                                            |                    |                |                   | 84.5            |

from 0.5 to 1 part per million. This error of reading, when calculated back to the original solution or to the dry soils, gives as great an error as the closer readings of the more dilute solutions, so this line of study was not carried further.

In addition to this study conducted with known solutions, the method has also been applied to the soil samples sent out this year by the Association of Official Agricultural Chemists. The results thus obtained are given in the following table, with results obtained by the standard methods for determining phosphoric acid. The average total reading in scale divisions and the corrections on each sample, also in scale divisions, are given in the table. These corrections are for original colour of the liquid and for filter-paper.

TABLE IX.—Phosphoric Acid Soluble in Distilled Water. Parts per Million of Dry Soil. 400 c.c. of Solution used for the Determinations.

| No. | Average total scale readings. | Correction in scale readings. | Reading due to $P_2O_5$ . | $P_2O_5$ colorimetric. | $P_2O_5$ volumetric. |
|-----|-------------------------------|-------------------------------|---------------------------|------------------------|----------------------|
| 2   | 13.5                          | 1.5                           | 12.0                      | 1.50                   | 1.33                 |
| 3   | 10.0                          | 1.1                           | 8.5                       | 1.06                   | 0.64                 |
| 4   | 10.0                          | 1.5                           | 7.5                       | 0.94                   | 1.26                 |
| 5   | 10.0                          | 3.0                           | 7.0                       | 0.87                   | 0.99                 |
| 6   | 48.5                          | 5.5                           | 42.0                      | 5.60                   | 5.10                 |
| 7   | 23.0                          | 2.5                           | 20.5                      | 2.56                   | 2.63                 |
| 8   | 20.0                          | 3.0                           | 17.0                      | 2.12                   | 1.95                 |

The results given are all that the writer has obtained on soils. No determinations have been rejected.

#### Precautions.

The method is so delicate (one scale division means one-half of one part per million on basis of dry soil when the

\* In solutions containing sufficient base to form normal phosphates with all the phosphoric acid, the addition of magnesium nitrate appears to be unnecessary.

† Or the organic matter may be destroyed by treating with aqua regia in the presence of sufficient base to prevent loss of phosphoric acid.

solution is prepared by treating 1 part of soil with 5 parts of water and taking 50 c.c. for the determination), and as has been shown there are so many points at which errors may be introduced, that a final word as to the precautions to be observed in handling it may be given with propriety.

The sodium phosphate ( $\text{Na}_2\text{HPO}_4 + 12\text{H}_2\text{O}$ ) from which the standard is made must be practically pure and be free from silica and iron. It is convenient to make a solution containing 100 parts phosphorus pentoxide per litre, and then dilute this to 10 parts per litre for the reading standard. The greatest care should be used in making this standard, as it is here where errors count. Ten c.c. of the 100 parts per litre solution should be run into a standardised 100 c.c. flask from an accurately graduated burette. Make up to a volume at 20–25° C. with distilled water (free from  $\text{SiO}_2 + \text{P}_2\text{O}_5$ ), add 10 c.c. of nitric acid (1.07) and 8 c.c. of ammonium molybdate, and mix thoroughly. This is the standard with which the unknown solutions are to be compared, and it should be made fresh each day.

All reagents, including distilled water, must be kept in Jena glass-ware, tested from time to time, and made fresh when a mixture of them shows colour after standing some time.

As the colorimetric tubes may have a slight colour themselves, each should be tested, and its reading carefully established. This may be done by filling the tubes with distilled water and reading them with a 1 part per million standard.

The standard and the unknown solution must contain, in equal volumes, equal amounts of reagents, and be at sensibly the same temperature.

The solution must contain less than 20 parts per million of iron (Fe).

A correction must be established for each package of filter-paper used. This correction is small, but not to be neglected on S. and S. No. 590 paper.

When the solution contains much lime or magnesia it is best to make two evaporations and filtrations before comparing.

Observing all precautions and making all corrections there is a maximum error of  $\pm 2$  scale divisions (each scale division = 1 c.c.) in the reading. When the soil solution is made by treating 1 part of soil with 5 parts of water, and 50 c.c. are taken for the determination, the error on the dry soil is  $\pm 2$  parts per million; when 100 c.c. are taken for the determination the error is  $\pm 1$  part per million, so that the working errors may only be neglected when at least 200 c.c. of solution are taken for the determination.

## PRECIPITATION AND SEPARATION BY WEAK ORGANIC BASES.\*

By E. T. ALLEN.

(Concluded from p. 89).

### Saponification of Methyl Acetate by Phenylhydrazine Hydrochloride at 40° C.

COMPARING the following results with those for aniline hydrochloride, we note that there is an acceleration in the rate of saponification in both cases. With aniline hydrochloride, this is comparatively small; with the phenylhydrazine compound, considerable. If the periods of time had been successive instead of overlapping, the acceleration would be seen to be actually greater than the figures indicate. If we compare the values for  $a$ , we have  $a = \text{N}/10$  hydrochloric acid, 7.4;  $\text{N}/10$  aniline salt, 7.49;  $\text{N}/10$  phenylhydrazine salt, 9.12.

|              |                      |            |
|--------------|----------------------|------------|
| $a_0 = 1.78$ | $a_{\infty} = 10.90$ | $a = 9.12$ |
| $t_1 = 1330$ | $x_1 = 0.37$         |            |
| $t_2 = 2419$ | $x_2 = 0.57$         |            |
| $t_3 = 4290$ | $x_3 = 1.55$         |            |
| $t_4 = 5683$ | $x_4 = 2.33$         |            |
| $t_5 = 6960$ | $x_5 = 3.02$         |            |

\* From the Journal of the American Chemical Society, xxv., No. 4.

$$K = 1.67 \times 10^{-4} \times \frac{26}{25} = 1.73 \times 10^{-4} + 1.22 \times 10^{-4} = 1.4 \text{ p. c.}$$

$$K = 1.44 \times 10^{-4} \times \frac{26}{25} = 1.50 \times 10^{-4} + 1.22 \times 10^{-4} = 1.2 \text{ p. c.}$$

$$K = 2.38 \times 10^{-4} \times \frac{26}{25} = 2.47 \times 10^{-4} + 1.22 \times 10^{-4} = 2.0 \text{ p. c.}$$

$$K = 2.89 \times 10^{-4} \times \frac{26}{25} = 3.00 \times 10^{-4} + 1.22 \times 10^{-4} = 2.5 \text{ p. c.}$$

$$K = 3.27 \times 10^{-4} \times \frac{26}{25} = 3.40 \times 10^{-4} + 1.22 \times 10^{-4} = 2.8 \text{ p. c.}$$

This means that, by some secondary reaction, more acid has been formed than the methyl acetate could furnish. On this account it seems more rational to calculate the percentages hydrolysed from the earlier periods of the process. This leads us to the approximate values 1.5 per cent for the phenylhydrazine salt and 2.0 per cent for the aniline compound. Many other experiments on the saponification of methyl acetate were made with other preparations of the salts, but since all led to the same conclusions, and the table given above contains the results obtained under the most carefully regulated conditions, no others have been quoted.

In calculating the values of  $K = \frac{1}{t} \log_e \frac{a}{a-x}$  for the phenylhydrazine salt,  $a$  was taken equal to 7.4.

A similar case of acceleration in the saponification of methyl acetate was noticed by Ley (*Zeit. Phys. Chem.*, xxx., 231) when he employed solutions of aluminum and lanthanum chlorides.

He attributed the acceleration to a secondary action between the chloride and acetic acid, setting free the more highly dissociated hydrochloric acid.

If we compare aniline and phenylhydrazine in regard to their power to precipitate the weak inorganic bases, it appears that their strength is nearly the same, but that of aniline is slightly less. (See Table F).

The precipitations were all made in the cold, with solutions containing about 1 m.grm. of metallic oxide per cubic centimetre, and all were slightly acid. 15 c.c. of each solution and about 1 c.c. of the reagent, a large excess, were taken for each test. Where any difference is found in the behaviour of the two bases, it is seen that phenylhydrazine precipitates more rapidly; and in one instance, beryllium sulphate, it precipitates where aniline has no action. That both reagents bring down the same precipitates one can scarcely doubt. They all closely resemble the hydroxides (or basic salts) formed by ammonia, being flocculent and white, or nearly so, when the reagents are free from colour and the solutions are dilute. The colour of phenylhydrazine is partly borne down by the precipitates. I have noticed that solutions containing free nitric acid in some quantity form yellow or brown products, which are probably oxidation products, and which colour the precipitates. The addition products which are formed by the action of the same bases on the chlorides of zinc, cadmium, and mercury, and by phenylhydrazine on the chlorides of cobalt and nickel as well, are entirely different in appearance; in fact, they are crystalline.

Regarding the difference in the behaviour of the nitrates and sulphates of the same elements, too little has been done to draw conclusions, but it will be noticed that where any difference exists, the sulphates precipitate more rapidly. The chlorides have not been systematically compared with the sulphates, but enough has been done to show that they behave similarly to the nitrates, in precipitating less rapidly and sometimes less completely than the sulphates. This may be due to the formation of basic salts in the case of the sulphates, precipitates which possess a different solubility from the hydroxides, or perhaps the sulphates of the organic bases are less dissociated than the chlorides,

TABLE F.

| Salt.                                              | Action of aniline.               | Action of phenylhydrazine.                                             |
|----------------------------------------------------|----------------------------------|------------------------------------------------------------------------|
| Be(NO <sub>3</sub> ) <sub>2</sub> ..               | Trace of precipitate .. ..       | Same as with aniline.                                                  |
| BeSO <sub>4</sub> ..                               | No precipitate .. ..             | Heavy precipitate, though not complete.                                |
| Al(NO <sub>3</sub> ) <sub>3</sub> ..               | Precipitation slow, but complete | Complete and much more rapid precipitation.                            |
| Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .. | Complete precipitation .. ..     | Complete precipitation.                                                |
| Cr(NO <sub>3</sub> ) <sub>3</sub> ..               | Complete, but slow precipitation | Complete and rapid precipitation.                                      |
| Cr <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .. | Complete precipitation .. ..     | Complete precipitation.                                                |
| Ti(NO <sub>3</sub> ) <sub>4</sub> ..               | " " .. ..                        | " "                                                                    |
| Ti(SO <sub>4</sub> ) <sub>2</sub> ..               | " " .. ..                        | " "                                                                    |
| Zr(NO <sub>3</sub> ) <sub>4</sub> ..               | Precipitation complete, but slow | Precipitation complete, and considerably more rapid than with aniline. |
| Zr(SO <sub>4</sub> ) <sub>2</sub> ..               | Complete precipitation .. ..     | Complete precipitation.                                                |

#### Concentration of Free Acid in the Solutions Precipitated by Organic Bases.

The solutions in which the previously described separations were made are too complicated in composition to deal with directly. All contained an unmeasured quantity of ammonium salt, while some contained also several different acids. A few experiments were therefore carried out under simplified conditions.

**Experiment 1.**—100 c.c. titanium sulphate containing 0.0044 grm. titanium dioxide were precipitated with ammonia, and the washed precipitate was transferred by a jet of water to an empty beaker. A measured quantity of hydrochloric acid, greater than that theoretically demanded, was then introduced. After warming and stirring, a large part of the precipitate remaining undissolved, an excess of phenylhydrazine was poured in. The precipitate was then filtered, while the filtrate was carefully tested for traces of titanium. None was found. It is evident that we have here data which, with the value of the hydrolytic constant of phenylhydrazine hydrochloride will enable us to calculate the quantity of free acid present. For if  $m$  equals the number of molecules of the phenylhydrazine salt,  $l$  equals the number of molecules of excess of base,  $v$  equals the volume of the solution in litres,  $k$  equals the hydrolytic constant, and  $a$  equals the degree of dissociation,—

$$k = \frac{a(m+l)}{v(1-a)} \text{ and } a = -\frac{kv+l}{2m} + \sqrt{\frac{kv}{m} + \left(\frac{kv+l}{2m}\right)^2}.$$

Total weight of acid = 0.238.

Phenylhydrazine required =  $0.238 \times \frac{108}{36.5} = 0.704$  grm.

= 0.006521 grm.-molecule.

Phenylhydrazine used = 1 c.c. = 1.09 grm.

Phenylhydrazine in excess =  $1.09 - 0.704 = 0.386$  grm.  
= 0.003575 grm.-molecule.

$m = 0.006521$  Hence  $a = 0.001$ , i.e., we have 0.006521  
 $l = 0.003575 \times 0.001 \times 36.5 = 0.2$  m.grm. of free  
 $k = 0.00002$  acid in 100 c.c.  
 $v = 0.2$  litre.

**Experiment 2.**—In this experiment all the conditions were the same as in Experiment 1, except that only 0.8 c.c. phenylhydrazine was used.

$m = 0.006521$   $a = 0.0027$ , i.e., we have 0.60 m.grm. of  
 $l = 0.001555$  acid in 200 c.c.  
 $k = 0.00002$   
 $v = 0.2$  litre.

**Experiment 3.**—To a quantity of freshly precipitated and washed alumina equivalent to 0.1257 grm. Al<sub>2</sub>O<sub>3</sub>, was added a very slight excess of hydrochloric acid, viz., 0.280 grm.; all the precipitate dissolved. At a boiling temperature, 2 c.c. phenylhydrazine gave a practically complete precipitation, but the precipitate was very slimy, and the conditions were not adapted to practical work. The excess of phenylhydrazine here is quite large, but other experiments with a smaller excess failed to precipitate the alumina completely.

Here  $m = 0.007672$  grm.-molecule.  
 $l = 0.01251$  grm.-molecule.  
 $k = 0.00000$   
 $v = 0.1$

Hence  $a = 0.00008$  and the free acid =  $0.007672 \times 0.00008 \times 36.5 = 0.02$  milligram. per 100 c.c.

The concentrations of free acid in the above cases were probably a little larger than those in the solutions with which the practical work was done, for in the latter cases there was an ammonium salt which must have tended to reduce dissociation.

It is quite certain that titanium hydroxide can be quantitatively precipitated in solutions more strongly acid, but we have here, no doubt, about the same amount of free acid as in the practical work.

There is some reason to believe that the calculated values given above are somewhat too small, for I found that blue litmus-paper, while it was decidedly reddened by the solutions we have just considered, gave hardly any reaction with solutions prepared by adding the calculated amount of free acid to 100 c.c. distilled water.\*

I am unable to explain this discrepancy. Possibly the value  $2 \times 10^{-5}$  for the hydrolytic constant of phenylhydrazine hydrochloride is too small, though one would be inclined to suspect that the method used would give high rather than low results. At all events it appears probable, in view of all the facts, that the concentration of free acid in the solutions we have been considering is not greater than a few milligrams. in 100 c.c.

#### Summary.

1. The weak organic bases, such as show little or no alkaline reaction with indicators, on account of the hydrolysis of their salts, can, of course, never completely neutralise the acid of a metallic salt. They therefore cannot precipitate the strong metallic bases, but only the weak ones, which are practically insoluble in very dilute acid. The precipitate may be either the hydroxide or a basic salt. All the analytical separations with these weak bases, which have been devised thus far, seem to involve this principle. The strong reducing power of phenylhydrazine gives it a particular advantage in certain cases.

2. The work described in this paper was done with phenylhydrazine and aniline. These two bases are of about the same strength, aniline being slightly weaker. This conclusion was arrived at by the study of the saponification of methyl acetate by their hydrochlorides, and also by their power to precipitate metallic hydroxides. The saponification constants in N/10 solutions at 40° C. were found to be  $2.5 \times 10^{-5}$  for the aniline salt, and  $1.7 \times 10^{-5}$  for the phenylhydrazine salt. These numbers lead us to the values  $4 \times 10^{-5}$  and  $2 \times 10^{-5}$ , respectively, for the hydrolytic constants. This means that about 2 per cent of the former and 1.50 per cent of the latter are decomposed into free base and free acid, under the conditions of dilution and temperature stated above.

3. The concentration of free acid in solutions from which the metallic hydroxides were separated must be quite small. The calculated values amounted to but a few tenths of a milligram. for such volumes as 100 to 200 c.c. There is some reason to think these values are too small, but it is not likely that they reach above a few milligrams. in the above volumes.

4. Aniline quantitatively precipitates the quadrivalent

\* A sensitive litmus tincture, however, reacted strongly with these solutions.

and weakly basic elements, titanium, zirconium, cerium, and thorium, as well as the trivalent elements  $\text{Fe}^{+++}$ ,  $\text{Al}$ , and  $\text{Cr}^{+++}$  under certain conditions, from dilute and slightly acid solutions. The solutions may be chlorides, nitrates, or sulphates. The same statements apply to phenylhydrazine, except that ceric and ferric salts are reduced by this reagent to salts of comparatively strong bases which are precipitated incompletely or not at all.

Zinc, cadmium, mercury, cobalt, and nickel, when sufficiently concentrated, form difficultly soluble addition products with phenylhydrazine. With aniline, also zinc, cadmium, and mercury give similar compounds. The strongly basic elements, magnesium, barium, calcium, strontium, manganese, and ferrous iron are not precipitated (*Journ. Am. Chem. Soc.*, xxi., 779).

Beryllium, when present alone, is not precipitated by aniline, nor by phenylhydrazine, except from sulphate solutions. Actual separations were worked out as follows:—Titanium and zirconium from iron; titanium, zirconium, and thorium from beryllium. The separation of aluminum from iron proposed by Campbell and Hess was studied more closely. A double precipitation is advisable in all these separations. The separations from ferrous iron depend upon the reducing power of phenylhydrazine as well as its weakly basic nature. Aniline cannot be substituted for it here, but all the separations from beryllium may be done equally well with aniline.

5. Phenylhydrazine will accurately separate minute quantities of alumina (and probably also the weaker bases) from large masses of iron.

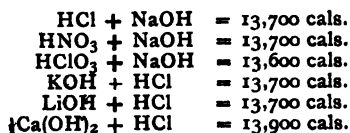
## WHAT PHYSICAL CHEMISTRY HAS DONE FOR CHEMISTRY.\*

By HARRY C. JONES.

(Concluded from p. 91).

*Heat of Neutralisation.*—We have a means of testing this crucially by experiment. If the process of neutralisation is independent of the nature of the acid and the nature of the base, the amount of heat set free when any completely dissociated acid acts on any completely dissociated base in equivalent quantities, will be a constant.

The fact is that a constant amount of heat (13,700 cal.) is liberated when a grm.-equivalent weight of any completely dissociated acid is brought in contact with a grm.-equivalent weight of a completely dissociated base. A few examples will make this clear:—



If we vary the nature of the acid or the nature of the base, the amount of heat set free is a constant to within the limit of experimental error. Fact and theory are thus in perfect accord.

*Thermoneutrality of Salts.*—Another application of the theory of electrolytic dissociation in thermochemistry is interesting and important. It has long been known that when dilute solutions of neutral salts are mixed there is no thermal change—heat is neither liberated nor absorbed. This is the well-known law of the thermoneutrality of salts. While the fact was established beyond question, and various suggestions had been made to account for it, it was generally conceded that no one of these was at all satisfactory. The explanation to-day is very simple. Take the two salts, potassium chloride and sodium nitrate; a dilute, aqueous solution of potassium chloride contains only

potassium ions and chlorine ions; a dilute solution of sodium nitrate contains sodium ions and nitric ions.



When the two solutions are mixed we have in the mixture potassium and sodium cations and chlorine and nitric anions, and nothing else. In a word, we have exactly the same ions in the mixture as in the solutions before they were mixed. There being no chemical change, there should be no thermal change, and such is the fact. The law of the thermoneutrality of salts could have been predicted from the theory of electrolytic dissociation, had it not been discovered long before the theory was discovered.

*Colour of Solutions.*—Another class of chemical phenomena which had long attracted attention, but the significance of which was not clearly understood, was the colour of substances, especially when in solution. Take, as an example, the colour of the permanganates. The salts of permanganic acid with the alkalis and alkaline earths all have apparently the same colour, which is the colour of permanganic acid itself. This was tested quantitatively by Ostwald, who photographed the absorption spectra of these and other permanganates where the cation was colourless, and showed that they all had exactly the same absorption bands. We can see at once the explanation of this fact. Permanganic acid dissociates thus:—



The colour of the solution is not due to the hydrogen ions, since there are hydrogen ions in solutions of all acids, and solutions of most acids are colourless. The colour of the solution of permanganic acid must be due to the presence of the permanganic ion:—



The colour of the solution of any permanganate in which the cation is colourless, is due to the ion:—



This can be seen at once, when we remember that any permanganate can be represented by the formula:—



in which R is a metal which yields a colourless cation. This compound would dissociate thus:—



$\overset{+}{\text{R}}$  being colourless, the colour of the solution must depend upon the coloured ion:—



We thus see at once why all such salts of permanganic acid must have exactly the same colour.

Ostwald studied in all about 300 coloured compounds, and found the theory of electrolytic dissociation most helpful in explaining phenomena similar to the above.

*Modes of Ion Formation.*—We have spoken thus far of molecules breaking down into ions in the presence of a dissociating solvent:—



It must, however, not be concluded that this is the only way in which ions can be formed from molecules.

We may have one metal precipitated from a solution of one of its salts by another metal. When a bar of zinc is immersed in a solution of a copper salt, the zinc passes into solution and the copper is thrown out of solution. What

\* From the *Journal of the Franklin Institute*, December, 1903.

takes place here is a transference of the electrical charge from the copper to the zinc. The copper having lost its charge, passes out of solution, while the zinc, having become an ion, passes into solution. This is obviously a mode of ion formation.

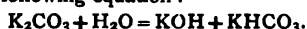
There are still other modes of ion formation. Take a bar of gold and dip it into chlorine water. Neither the gold nor the chlorine is in the ionic state before they come in contact. When they come in contact the gold becomes cationic and passes into solution, while the chlorine remains in solution, but passes from the molecular into the anionic condition. We have both cations and anions formed under conditions which are usually described as the solution of gold in chlorine water.

**Solutions of Metals in Acids.**—There are few chemical processes with which we are more familiar than the solution of metals in acids. Most metals dissolve in the strong acids, and from such solutions salts of the metals can be obtained on evaporation. This fact, which has been so long known, has only recently been satisfactorily explained. An acid dissociates, always yielding hydrogen ions, and anions whose nature depends upon the acid in question. This may be represented in general as follows:—

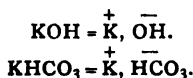


When such an acid is brought in contact with a metal, the hydrogen ions of the acid give up their charge to the metal, and escape as hydrogen gas. The metal, having taken the charge, passes into solution in the ionic state. This is a perfectly general process, independent of the nature of the acid and independent in general of the nature of the metal. In reality it is a mode of ion formation. An ion which holds its charge less firmly—hydrogen—giving it up to the metal which holds the charge more firmly, the hydrogen escaping in the gaseous condition, the metal passing into solution.

**Hydrolytic Dissociation.** Thus far the subject of electrolytic dissociation, or the breaking down of molecules into ions, has been considered. There is another kind of dissociation, which takes place in solutions of certain chemical compounds. When an alkali salt of a weak acid like carbonic acid is brought into the presence of water it shows an alkaline reaction. The fact has long been known, but no satisfactory explanation of it has been furnished. We have to-day a perfectly rational explanation of these and similar facts. There is at first a chemical reaction between the salt and the water, which takes place in the sense of the following equation:—



This is known as *hydrolytic dissociation*. As soon as the products of hydrolytic dissociation are formed they are electrolytically dissociated by the water. In the above case the following ions are formed:—



The hydroxyl ions from the dissociated water give the alkaline reaction, which is characteristic of such solutions.

We can thus see why the salts of weak acids react alkaline, and in a similar manner can understand why the salts of weak bases react acid. They are hydrolytically dissociated by water yielding the free acid, which then dissociates yielding hydrogen ions, which give the characteristic reaction to the solution.

**Faraday's Law the Basis of Valence.**—The lecturer then took up a discussion of Faraday's law as the basis of chemical valence. He pointed out that if we refer valence to the law of Faraday, we place it upon an exact physical basis. If we do not, the name valence is more or less indefinite, being used with a great variety of meanings.

**Importance of Energy Changes for Chemistry.**—The lecture concluded with a plea for the necessity of dealing with the energy changes which take place when substances react chemically. Hitherto we have regarded these energy

changes as subordinate, and have referred to chemical reactions as being accompanied by thermal change. This is confusing cause and effect. It would probably be much nearer the truth to say that thermal changes are accompanied by material transformations.

It was then pointed out that by studying material transformations alone we can never have an *exact science of chemistry*. This can be realised only by studying together with the products of the reaction, the thermal changes, the electrical changes, the light changes; in a word, the reciprocal transformations of intrinsic and other forms of energy.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 4, January 25, 1904.

**Fluochlorides, Fluobromides, and Fluoioidides of the Alkaline Earth Metals.**—Ed. Defaçon.—The author shows that when a mixture of manganous fluoride is treated at temperatures between 800° and 1400° with chloride, bromide, and iodide of an alkaline earth, the alkaline earth fluoride results, together with the chloride, bromide, and iodide of manganese, the quantity of the latter increasing in proportion as the quantity of manganese fluoride is increased. He also shows that manganese chloride, bromide, and iodide react in the same way on alkaline earth fluorides. It follows therefore that, in the fused liquid, an equilibrium must be produced for the two reversible reactions. The transformation of manganous fluoride into an alkaline earth fluoride is only total when the quantity of chloride, bromide, or iodide of manganese, as compared with the alkaline-earth chloride, bromide, or iodide, is very small; the fluochlorides, fluobromides, and fluoioidides cannot then be formed, since they are destroyed by the chloride, &c., during fusion. Alkaline earth fluorides are formed when the manganous fluoride is mixed with a large excess of alkaline earth chloride, bromide, or iodide. Fluochlorides, fluobromides, and fluoioidides of the alkaline earths are also formed when the alkaline earth halides are entirely transformed into manganous chloride, bromide, or iodide by employing the theoretical quantities demanded by the equation  $MnF_2 + 2XY_2 = XF_2 \cdot XY_2 + MnY_2$ ; X representing Ba, Sr, or Ca, Y representing Cl, Br, or I.

**Colour Reactions of Molybdic Acid.**—Emm. Pozzi-Escot.—When a few drops of a solution of tannin is added to a solution of molybdic acid an orange solution is formed, becoming cherry-red in strong solution and yellow in dilute solution. This colouration constitutes a very sensitive test for molybdic acid, even showing with 1/100,000 part of ammonium molybdate in solution.

**Electrolysis of Chloric Acid and Chlorates.**—André Brochet.—The author investigates the mechanism of the action of copper on chloric acid and copper chlorate, both with and without the use of electrolysis, and elucidates the reactions—really simple, but apparently complex—which take place during the electrolysis of chlorates of the alkaline and alkaline-earth metals with a copper anode.

**Trichlor-isopropyl Alcohol,  $Cl_3C-CH(OH)-CH_3$ .**—Louis Henry.—The author completes the series of the methylation of the chlor-ethyl alcohols, and prepares trichlor-isopropyl alcohol,  $Cl_3C-CH(OH)-CH_3$ , by the reaction of anhydrous chloral on the magnesium compound of methyl iodide in ether. The substance when purified is crystalline, melting at 50–51° and boiling at 161.8° under a pressure of 773 m.m. It is interesting to notice the variation in properties in the series of trichlor-alcohols containing  $C_2$ ,  $C_3$ , and  $C_4$ .

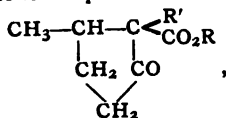


|                                                                                                                     | Fusion.    | Ebullition. |
|---------------------------------------------------------------------------------------------------------------------|------------|-------------|
| $\text{Cl}_3\text{C}-\text{CH}_2-\text{OH}$ .. ..                                                                   | $17^\circ$ | $151^\circ$ |
| $\text{Cl}_3\text{C}-\text{CH}-\text{OH}$ .. ..                                                                     | $50^\circ$ | $161^\circ$ |
| $\begin{array}{c} \text{CH}_3 \\   \\ \text{CH}_3 \\   \\ \text{Cl}_3\text{C}-\text{C}-\text{OH} \end{array}$ .. .. | $96^\circ$ | $167^\circ$ |

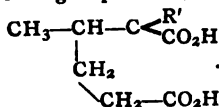
**Condensation of Acetylenic Ethers with Alcohols.**—Charles Moureu.—In a previous paper the author showed that methyl phenylpropionate, under the influence of sodium methylate in methylic alcohol solution, can fix 2 molecules of methylic alcohol, with formation of dimethylacetal of methyl benzoylacetate, by opening and completely saturating the acetylenic liaison, according to the equation—  
 $\text{C}_6\text{H}_5-\text{C}\equiv\text{C}-\text{CO}_2\text{CH}_3 + 2\text{CH}_3\text{OH} =$   
 $\text{C}_6\text{H}_5-\text{C}(\text{OCH}_3)_2-\text{CH}_2-\text{CO}_2\text{CH}_3$ .

This new reaction is now generalised, and the author studies from the same point of view the series of the acetylenic ethers.

**$\alpha$ -Substituted  $\beta$ -Methyladipic Acids.**—Marcel Desfontaines.—It is known that the  $\alpha$ -methylcyclopentanonecarboxylic ethers behave with regard to sodium alcoholates and the alcoholic iodides as camphocarboxylic ethers, giving rise to compounds of the form—



which, by a simple saponification with alcoholic potash, gives the corresponding adipic acids,—



The author prepares a series of these cyclic compounds and the corresponding di-substituted acids.

**Certain Derivatives of Tetramethyldiaminophenyl-oxanthranol.**—MM. Guyot and Stehling.—In a previous research one of the authors established the fact that tetramethyldiaminophenyl-oxanthranol can easily attach itself to a certain number of products of the aromatic series without elimination of water. The present authors make analogous condensations with anisol and phenetol.

## MEETINGS FOR THE WEEK.

- MONDAY, 29th**—Society of Arts, 8. (Cantor Lectures). "Modern Book Printing," by Charles T. Jacobi.
- TUESDAY, March 1st**—Royal Institution, 5. "Japanese Life and Character," by Ernest Foxwell, M.A.  
 Society of Arts, 4.30. "Nigeria," by Lady Lugard (Miss Flora L. Shaw).
- WEDNESDAY, 2nd**—Society of Arts, 8. "Physical Degeneration," by Robert Jones, M.D., &c.  
 Society of Public Analysts, 8. "Composition of Milk," by H. Droop Richmond, F.I.C. "Estimation of Morphine in Opium," by P. Schidrowitz, Ph.D. "Iodine Absorption as a Factor in the Examination of Otto of Roese," by F. Hudson-Cox, F.I.C., and W. H. Simmons.
- THURSDAY, 3rd**—Royal Institution, 5. "Electrical Methods of Measuring Temperature," by Prof. H. L. Callender, F.R.S.  
 Chemical, 8. "Chemical Dynamics of the Alkyl Iodides," by Miss K. A. Burke and F. G. Donnan. "Constitution of Phenolphthalein," by A. G. Green and A. G. Perkin. "A Ketohexahydrobenzole Acid," by W. H. Perkin, jun. "Photo-chemically Active Chlorine," by C. H. Burgess and D. L. Chapman.
- FRIDAY, 4th**—Royal Institution, 9. "Breathing in Living Things," by Prof. William Stirling, M.D., &c.
- SATURDAY, 5th**—Royal Institution, 3. "The Life and Work of Stokes," by Right Hon. Lord Rayleigh, F.R.S.

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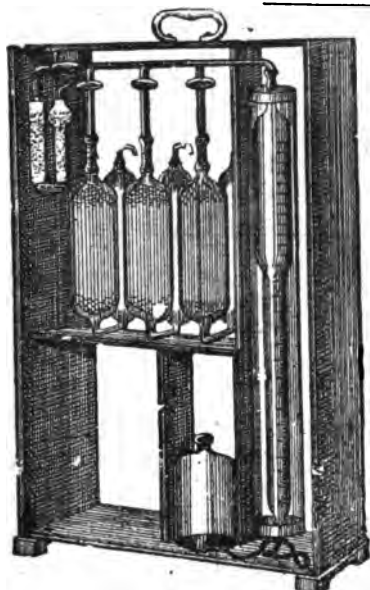
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### CONTENTS.

| ARTICLES:—                                                                                                                       | PAGE |
|----------------------------------------------------------------------------------------------------------------------------------|------|
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S. ....                                                 | 109  |
| Some Applications of the Theory of Electrolysis to the Separation of Metals from one another, by A. Hollard ....                 | 110  |
| Estimation of Persulphates, by C. Marie and L. J. Brunel ....                                                                    | 113  |
| Volumetric Estimation of the Alkaline Nitroprussiates and of the Soluble Salts of Cadmium, by MM. Fonsee-Diacon and Carquet .... | 114  |
| PROCEEDINGS OF SOCIETIES—                                                                                                        |      |
| CHEMICAL SOCIETY .....                                                                                                           | 114  |
| THE FARADAY SOCIETY .....                                                                                                        | 117  |
| CORRESPONDENCE.—Why are some Elements more Abundant than others? .....                                                           | 118  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                      | 119  |
| MISCELLANEOUS .....                                                                                                              | 120  |
| MEETINGS FOR THE WEEK .....                                                                                                      | 120  |

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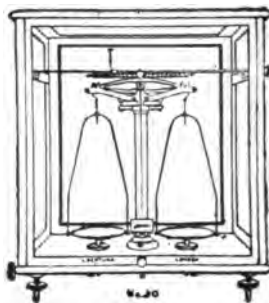
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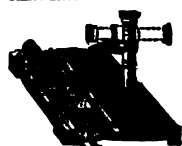
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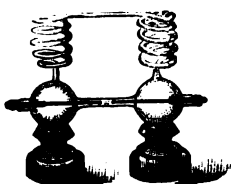
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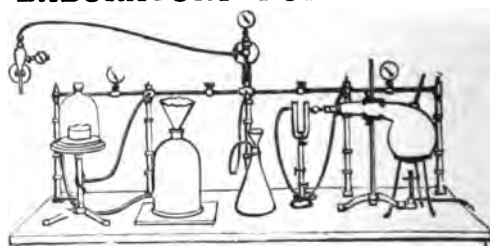
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2310.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.\*

By CHARLES BASKERVILLE, Ph.D., F.C.S.,  
Smith Professor of General Chemistry, University of North Carolina.

It is the sad duty of the retiring Chairman of this Section to chronicle the death of two members. One of them, James Francis Magee, B.S., University of Pennsylvania, 1887, devoted his life chiefly to commercial pursuits, in which he was most successful. He joined the Association at the fifty-first meeting, being one of our youngest. The other was H. Carrington Bolton, Columbia, 1862, (Ph.D., Göttingen, 1865), who, with the exception of four (Gibbs, Boye, Brush, and Hilgard), was the senior of the Section, having joined at the seventeenth meeting. I beg permission to quote from an article of his in the *American Chemist*, 1876, the year following his elevation to Fellowship in the Association, as it exemplified, in telling words, one of the great aims in his life, with the fruitful accomplishment of which you are familiar:—

"So rapid are the strides made by science in this progressive age, and so boundless is its range, that those who view its career from without find great difficulty in following its diverse and intricate pathways, while those who have secured a footing within the same road are often quite unable to keep pace with its fleet movements, and would fain retire from the unequal contest. It is not surprising, then, that those actually contributing to the advancement of science, pressing eagerly upward and onward, should neglect to look back upon the labours of those who precede them and should sometimes lose sight of the obligations which science owes to forgotten generations."—"Notes on the Early Literature of Chemistry—The Book of the Balance of Wisdom," New York Academy of Sciences, May 29, 1876.

His numerous contributions to and intimate knowledge of the history of chemistry, his gentle and generous sympathy, aided and stimulated many active in research or technical applications of chemistry. His monumental bibliographies put out by the Smithsonian Institution are masterpieces. The grief and keen regret of his loss are not confined to one nation.

On another occasion it has been the good fortune of him who has the honour of addressing you to-day to indicate that events of literary moment, governmental modifications, inventions, and forward stridings in science have apparently accommodated themselves to historical periods during the past century ("The Rare Earth Crusade—What it Portends, Scientifically and Technically," *Science*, N.S., xvii., 722; *CHEMICAL NEWS*, lxxxviii., 18). Striking novel facts and fancies, gleaned in the realm of inorganic chemistry, have crested not a few of the high waves of those human tides that beat against the coast of the untried and unknown.

The human mind knows by contrasts. For the day we have night, for the good there is evil. Where man would have a God, he also had a devil; for the true there is the false; the verified and unverified. The false may be true through ignorance; the true may be false in the light of new knowledge. Or, as Hegel put it, "Sein und das nicht Sein sind Nämliche."

Is matter continuous or discrete? argued the opposed schools of Grecian philosophy led by Leucippus, Democritus, and Epicurus, and dominated by Aristotle. Despite the clarity

of the statements of the Roman Lucretius,\* the atomic hypothesis received scant attention until the Seventeenth century of the Christian Era, when Galilei's experimental science assailed Aristotelian metaphysics, and demanded verification of the promises of that philosophy which had governed all the schools of Europe for two thousand years (see "The Atomic Theory," the Wilde Lecture, by F. W. Clarke, at Dalton Celebration, May, 1903; *CHEM. NEWS*, lxxxviii., 260). While Gassendi, Boyle, Descartes, Newton, perhaps Bosovich, Lavoisier, Swedenborg, Richter, Fischer, and Higgins had to do with our modern atomic theory, Dalton, one hundred years ago, "created a working tool of extraordinary power and usefulness" in the laws of definite and multiple proportions. As Clarke remarked (*loc. cit.*), "Between the atoms of Lucretius and the Daltonian atom the kinship is very remote." Although the lineage is direct, the work of Berzelius, Gmelin, and others,—the laws of Faraday, Gay Lussac, Avogadro, Dulong and Petit,—the reformations of Laurent and Gerhardt, but particularly Cannizzaro,—the systematisations of de Chancourtois, Newlands, Hinrichs, Mendeleeff, and Lothar Meyer,—the stereo-chemistry of van't Hoff and Le Bel, have imperialised the ideas of the Manchester philosopher, so that the conceptions of the conservative atomists of to-day are quite different from those at the beginning of the closed century.†

These have not come about solely through the additive labours of the *savants* mentioned, for they have been shaped quite as much by speculative and experimental opposition exemplified by Brodie ("Calculus of Chemical Operations," *Journ. Chem. Soc.*, 1866, xxi., 367; and his book "Ideal Chemistry," 1880) and Sterry Hunt (numerous papers summarised in "A New Basis for Chemistry," New York, 1887 and 1892, 4th edition).

In Graham's "Speculative Ideas respecting the Constitution of Matter" (*Proc. Roy. Soc.*, 1863) we have the conception that our supposed elements possess "one and the same ultimate or atomic molecule existing in different conditions of movement" (Venable, "The Definition of the Element," Vice-Presidential Address, Section C, A.A.A.S., Columbus Meeting, 1899). *Apropos*, we have the suggestion of F. W. Clarke, that the evolution of planets from nebulae, according to the hypothesis of Kant and Laplace, was accompanied by an evolution of the elements themselves ("Evolution and the Spectroscope," *Pop. Sc. M. Journ.*, 1873). Even Boyle—"the cautious and doubting Robert Boyle," as Humboldt said of him—was inclined to the belief that "all matter is compounded of one primordial substance—merely modifications of the *materia prima*."

The Daltonian ideas had scarcely reached adolescence before Prout (1815), giving heed to the figures concerned, would have all the elements compounded of hydrogen. The classical atomic mass values obtained by sympathetic Stas and the numerous investigations of those who followed him, with all the refinements human ingenuity has been able to devise, temporarily silenced such speculations, but not until Marignac had halved the unit, Dumas had quartered it, and Zängerle, as late as 1882, insisted upon the one-thousandth hydrogen atom.

The notion, like Banquo's ghost, will ever up, for if one may judge from the probability calculations of Mallet (*Phil. Trans.*, 1881, clxxi., 1003) and Strutt (*Phil. Mag.*, 6], i., 311), a profound truth underlies the now crude hypothesis.

Crookes (*CHEMICAL NEWS*, 1886, lv., 83), from observations made during prolonged and painstaking fractionations of certain of the rare earths, supported his previously

\* "Nature reserving these as seeds of things  
Permits in them no minish nor decay;  
They can't be fewer and they can't be less."

Again, of compounds,—

"Decay of some leaves others free to grow  
And thus the sum of things rests unimpaired."

—*Hor. II.*, 79.

† While I have examined much of the original literature, Venable's "History of the Periodic Law" has been most helpful. I have, furthermore, had the privilege of reading very carefully the manuscript of a work entitled "The Study of the Atom" (in press), by Dr. Venable.

\* Address of the Vice-President of Section C (Chemistry) of the American Association for the Advancement of Science, St. Louis Meeting, December 28, 1903.



announced "provisional hypothesis" as to the genesis of the elements from a hypothetical *protyle* which existed when the universe was without form and void. He designated those intermediate entities, like yttrium, gadolinium, and didymium, "meta-elements"—a species of compound radicals, as it were (Address before Chemical Section of the British Association, CHEMICAL NEWS, 1885, liv., 117). *Urstoff*, fire-mist, protyle, the ultra-gaseous form, the fourth state of matter, was condensed by a process analogous to cooling; in short, the elements were created (Crookes, Royal Society, June 10, 1880). The rate of the cooling and irregular condensation produced "the atavism of the elements," and this caused the formation of the natural families of the periodic system. Marignac, criticising this hypothesis, states (*Archives des Sciences Physiques et Naturelles*, xvii., 5; CHEMICAL NEWS, lvi., 39):—"I have always admitted\* the impossibility of accounting for the curious relations which are manifested between the atomic weights of the elements, except by the hypothesis by a general method of formation according to definite though unknown laws; even when these relations have the character of general and absolute laws."

Further, "I do not the less acknowledge that the effect of constant association of these elements is one of the strongest proofs that can be found of the community of their origin. Besides, it is not an isolated fact; we can find other examples, such as the habitual association in minerals of tantalum, niobium, and titanium."

Sir John Herschel thought that all the atoms were alike, and the elements, as we know them, "have the stamp of the manufactured article."

Hartley, this year, says (Address before the Chemical Section, British Association, Southport Meeting, Sept., 1903; CHEMICAL NEWS, lxxxviii., 154):—"It is more than twenty years since the study of homology in spectra led me to the conviction that the chemical atoms are not the ultimate particles of matter, and that they have a complex constitution."

The peculiar discharge from the negative electrode of a vacuum tube was investigated many years ago by Hittorff and Crookes, who arrived at the conclusion that it was composed of streams of charged particles. All are familiar with the very recent proposed "electrons" and "corpuscles" resulting from the beautiful physical researches of Lodge and J. J. Thomson. These appear to have caused a trembling in the belief of many in the immutability of the atom, and the complete abandonment of the atom is seriously discussed by others.

"If the electrons of all elements are exactly alike—or, in other words, if there is but one matter, just as there is but one force—and if the elements be but the various manifestations of that one matter, due to a different orbital arrangement of the electrons, it would seem that we are fast returning to the conceptions of the Middle Age alchemist. The transmutations of metals involves but the modification of the arrangement of the electrons." Such efforts as Fittica's ("Black Phosphorus, or Conversion of Phosphorus into Arsenic," CHEMICAL NEWS, lxxxi., 257; lxxxi., 166) should not be treated with scorn, but given the careful examination and merited consideration as Winkler gave his (*Berichte*, xxxiii., 10; CHEMICAL NEWS, lxxxi., 305). Science should thus ever be "a foe of raw haste, half-sister to delay" (Van Dyke in "The Ruling Passion").

Although by chemical means so far we have been unable to break up the atom, apparently electrical energy—in the form of cathode rays, for example—follows the grain of atomic structure. Some advanced thinkers look upon the atoms as disembodied charges of electricity. Ostwald has taught it. Electric charges are known only as united to matter, yet Johnstone Stoney and Larmor have speculated on the properties of such charges isolated. "Such a charge is inertia, even though attached to no matter, and the increase of inertia of a body due to electrification has been calculated by both Thomson and Oliver Heaviside, the

conception accordingly being advanced that all inertia is electrical, and that matter, as we know it, is built up of interlocked positive and negative electrons. If it were possible in any mass of matter to separate these electrons, then matter would disappear, and there would remain merely two enormous charges of electricity." We are aware of phenomena attributed to the negative electrons; we await anxiously the announcement of the positive electrons. But here the water is deep, and one may not swim too well.

We do know, however, as A. A. Noyes says ("General Principles of Physical Science," 1902, p. 13), that "there exists in the universe some thing or things other than matter, which, by association with it, gives rise to the changes in properties which bodies exhibit, and gives them power of producing changes in the properties of other bodies." Further (p. 15), "... matter is that which gives rise to the localisation of the complex of properties which certain portions of space exhibit. Even though, on the one hand, it must be admitted that the existence of matter is inferred only from various energy manifestations which bodies exhibit, it must be acknowledged, on the other, that there are no manifestations of energy except those which are associated with the manifestations of it that have led to the adoption of the concept of matter; in a word, the two assumed entities—matter and energy—are indissolubly connected in our experience." Thus, as Dumas said, "Hypotheses are the crutches of science, to be thrown away at the proper time."

I have dared to sketch these conceptions in a few bold outlines, for—

"We can't enumerate them all!  
In every land and age have they  
With honest zeal been toiling on,  
To turn our darkness into day."\*

(To be continued).

## SOME APPLICATIONS OF THE THEORY OF ELECTROLYSIS TO THE SEPARATION OF METALS FROM ONE ANOTHER.†

By A. HOLLARD.

In the present study I have co-ordinated a certain number of facts and experiments already published here and there in such a way as to bring them into line and show what philosophic bearing they have on one another. It thus clears the way for the presentation of an actual theory of electrolytic analysis that has not up to the present been put forward. The only principle involved hitherto in electrolytic analysis for the separation of elements is that which is based on the successive separation of the metals by gradual increase of the potential across the electrodes, each metal depositing itself at a potential spoken of as the potential proper to the metal.

Strictly speaking, this principle is hardly directly applicable. Indeed, it supposes the constant use of low potentials, and consequently small intensities, thereby prolonging the duration of the experiment. In fact, the only applications of this principle which have been made are the separations with very weak currents, of copper and silver, of silver and bismuth, and of mercury and bismuth, that is of metals whose polarisation potentials are lower than that of hydrogen.

As to metals with polarisation potentials higher than that of hydrogen (zinc, cadmium, iron, cobalt, nickel, tin, lead), they cannot be separated according to this same principle. In fact, the current, already very weak, which precipitates one of the metals, separates hydrogen from the bath just as much, and then the fraction of the current utilised for the deposit of the metal is so small, especially towards the end of the electrolysis, when the concentration

\* Remarks made in 1860–65, after publication of Stas's "Researches on Atomic Weights," *Archives*, ix., 102, 24–376.

\* Aikens' poem at Priestley centennial, *American Chemist*, 1875, 23.  
† A Paper read before the Faraday Society, February 2, 1904.

of the metal ions becomes very low (Nernst's law), that the deposition of the metal is never complete. Thus zinc and nickel, which have a relatively large potential difference (0.54), have not been separated by us, when we used a slightly ammoniacal solution of the sulphates with a current of 0.01 ampère, the zinc always depositing itself with the nickel. We ought to work with a current of less than 0.01 ampère, seeing that a fraction only is utilised, but this would make the operation interminable.

Recognising that the foregoing principle is entirely insufficient, we have sought for other applications of the theory of electrolysis permitting of effectual analytical separations. The applications we have found will be ranged under three headings.

- I. Reduction of the resistance of the bath by suppressing the formation of gas at the anode.
- II. Influence of the nature of the cathode.
- III. Formation of complex salts. (This application has already been studied, especially by Frendenberg).

#### I Reduction of the Resistance of the Bath by suppressing the Formation of Gas at the Anode.

We have shown above that the metals whose polarisation potentials are higher than that of hydrogen cannot be separated from one another by gradual increase of the E.M.F., on account of the extremely small fraction of the current used to precipitate the metal. We have been able, however, considerably to increase the current by diminishing the resistance of the bath, and that in two ways.

1. By suppressing the liberation of oxygen at the anode.
2. By the use of soluble anodes.

1. To suppress the liberation of oxygen at the anode (or at least greatly to diminish it), we introduce a reducing agent into the bath, such as sulphurous acid, which prevents the liberation of oxygen gas by combining with it. This suppression of gas greatly augments the conductivity of the bath; for the same potential applied to the bath the current which passes is much more intense, and permits the precipitation of one of the metals on the cathode in a relatively short time.

Let us take a mixture of zinc and nickel, which cannot be separated by the application of the first principle; the sulphates of the metals are treated with sulphate of ammonia (10 grms.), sulphate of magnesium (5 grms.), 5 c.c. of a saturated solution of sulphurous anhydride, finally an excess of 25 c.c. of ammonia (sp. gr. 0.924). This is diluted to 300 c.c. and electrolysed at a temperature of about 90°, with a current of 0.1 ampère. The electrodes we used are shown in Fig. 1, the cathode being of platinum gauze, and sand-blasted.†

At the end of four hours at the most, for quantities of nickel which do not exceed 0.25 gm., 1–2 c.c. of the bath should not be blackened by ammonium sulphide. The electrolysis is allowed to continue for an hour, then the cathode is removed.

#### Experimental Results.

| Quantities weighed. |      | Nickel deposited |
|---------------------|------|------------------|
| Ni.                 | Zn.  |                  |
| 0.2500              | 0.05 | 0.2508           |
| 0.2500              | 0.1  | 0.2494           |
| 0.2500              | 0.25 | 0.2517           |
| 0.2500              | 0.5  | 0.2500           |
| 0.2500              | 0.5  | 0.2506           |
| 0.2500              | 1.0  | 0.2501           |
| 0.1000              | 0.1  | 0.0969           |
| 0.1000              | 0.5  | 0.0963           |
| 0.1000              | 1.0  | 0.0973           |

It is very important to adhere to the proportions of the reagents indicated, otherwise the separation may fail.

† It is important that the temperature should never fall below this value.

† These can be procured from des Moutis, of London and Paris, and Poulenç, of Paris.

Thus an excess of sulphurous acid leads to the formation of zinc sulphide.

This portion of the work was carried out with Mons. Bertiaux.

2. The use of a soluble anode to avoid the liberation of oxygen at the anode is less simple than the above procedure. On the other hand, the soluble anode in dissolving produces sufficient electric current to electrolyse the bath, and render the use of an external source of electricity unnecessary.

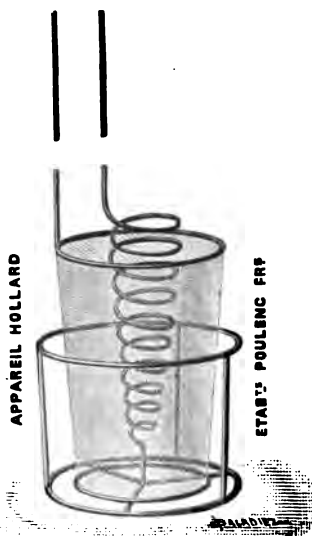


FIG. 1.

The metal selected for the anode should be of such a nature that it can replace in the solution the metal that is to be separated, and that only. In order that this may not be precipitated on the anode, but only on the platinum cathode, the anode must be immersed, not in the bath, B, itself (Fig. 2), but in the solution A containing an indifferent salt (salt of an alkali or an alkaline earth) separated from the bath B by a membrane of vegetable parchment.

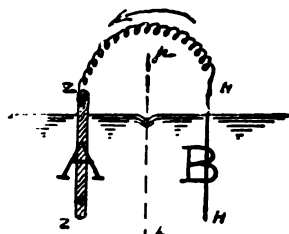


FIG. 2.

If it is required to separate nickel from zinc, we make use of a zinc anode. We take a vessel separated into two cells by a membrane *p p* of vegetable parchment (paper treated with sulphuric acid). We place in one of the cells, B, the solution containing the nickel and the zinc, and in the other, A, a salt solution, which must be a conductor of the current—a solution of magnesium sulphate serves very well. The zinc sheet intended to precipitate the nickel is immersed, not in the solution B containing the nickel, but in the magnesium sulphate solution. In the solution B, a sheet of platinum *h h* is placed. Finally, the platinum and zinc are connected by an external copper wire. We thus make a reversible cell of two liquids of the Daniell type, in which the current goes from the platinum to the zinc by an

external conductor, and from the zinc to the platinum in the interior of the liquid.

We will therefore examine an actual electrolysis of a nickel salt, giving a deposit of pure nickel on the platinum sheet :—

In practice we proceed as follows :—

The nickel and zinc in the state of soluble sulphates are contained in a Bohemian beaker (650 c.c. capacity, 7 c.m. diameter). This solution should contain an excess of 20 c.c. ammonia, 22° Bé., and 100 grms. of anhydrous ammonium sulphate, and should occupy a volume of 250 c.c.

Obviously this liquid is a good conductor of the current. We put into it our electrode of platinum gauze (Fig. 3). A glass tube 55 m.m. internal diameter, closed at the base by a membrane of vegetable parchment, is immersed in the solution of nickel and zinc in the beaker, so that the membrane is as near as possible to the electrode without, however, touching it. A solution of magnesium sulphate,

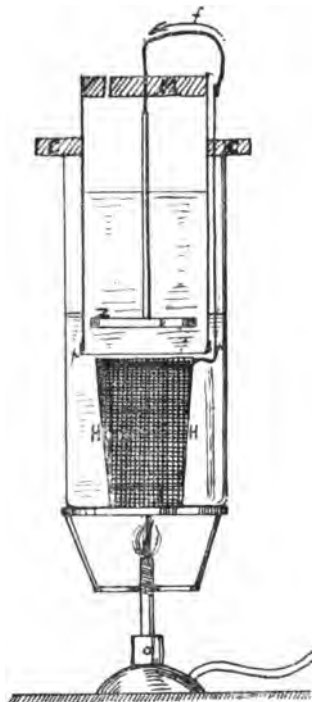


FIG. 3.

250 grms. to the litre, is poured into the tube to fill it to a depth of about 70 m.m. This strength of magnesium sulphate solution possesses the maximum conductivity. The choice of this salt as conductor for the inner cell is that it is one of the few salts which have no chemical action on zinc.

Finally, an amalgamated zinc disc, 5 c.m. diameter, attached to a rod of copper insulated by caoutchouc, is immersed in this liquid. The copper rod is connected externally to the platinum electrode. The zinc disc is pierced with several holes to facilitate the circulation of the inner cell liquid, and placed 15—20 m.m. above the parchment.

During the whole of the experiment, the contents of the outer cell are heated nearly to boiling (about 95°). At the end of several hours the nickel is completely deposited on the electrode as a fine, strongly adherent metallic layer. The electrode is withdrawn, swilled with water, then plunged into distilled water for ten minutes to complete the washing, finally dried and weighed.

### Experimental Results.

| Quantities taken. |     | Nickel deposited.         |
|-------------------|-----|---------------------------|
| Ni.               | Zn. |                           |
| 1 .. .. 0'2000    | 1   | 0'2026                    |
| 2 .. .. 0'2000    | 0'5 | 0'1995                    |
| 3 .. .. 0'2500    | 1   | 0'2499                    |
| 4 .. .. 0'2500    | 1   | 0'2525                    |
| 5 .. .. 0'5000    | 0'0 | 0'4989                    |
| 6 .. .. 0'4500    | 1   | 0'4506                    |
| 7 .. .. 0'5000    | 1   | 0'5048                    |
|                   |     | (contained a little zinc) |
| 8 .. .. 0'0500    | 1   | 0'0524                    |

One sees from the above table that the method can be applied to varying proportions of zinc and nickel. On the other hand, the mixture should not contain more than 1 gm. of zinc or 0'45 gm. of nickel, otherwise the simultaneous deposition of zinc becomes possible.

Experiment 7 shows that already with 0'5 gm. of nickel and 1 gm. of zinc, there is a deposition of zinc. When there is no zinc in solution at least 0'5 gm. pure nickel can be deposited (Experiment 5).

These limits as to the quantities of nickel and zinc find their explanation in the theory of the method we are about to give. This theory is none other than that of reversible cells.

The solution-pressure  $P$  of the precipitating metal\* (in this case zinc) being higher than the solution-pressure  $P_1$  of the precipitated metal† (here nickel), a positive E.M.F. is established between the two metals, according to Nernst's formula :—

$$(1) \epsilon = \frac{K}{v} \log \frac{P}{C} - \frac{K}{v_1} \log \frac{P_1}{C_1};$$

$K$  is a constant for a definite temperature,  $v$  and  $v_1$  are the valencies of the precipitating and precipitated metals,  $C$  and  $C_1$  the concentrations of the two metals.†

One sees, according to this formula, that when the concentration  $C$  of the zinc in the inner cell increases (consequent upon the deposition of nickel on the platinum electrode)  $\epsilon$  will diminish. It is necessary then that there should not be too much nickel to deposit; 0'45 gm. can, however, be easily deposited. According to the same formula, if the concentration of the nickel diminishes,  $\epsilon$  tends towards zero, which means that the nickel can never be completely deposited. This would indeed happen if we were working with a solution of the normal sulphate of nickel, but we work with a solution containing a large excess of ammonia; that is, a solution containing a large concentration of the ions  $\text{NH}_4^+$ ; thus  $C_1$  does not represent the concentration of the nickel ions, but a mean of the concentration of the ions  $\text{Ni}^{++}$  and  $\text{NH}_4^+$ . This ammonium gives rise to a disengagement of hydrogen during the electrolysis.

Therefore, when one would generalise this method for other separations than that of nickel and zinc, care must be taken to put into the solution an excess of ammonia or acid, or any other substance capable of forming hydrogen ions, or ions playing the same part.

In reality, the phenomenon is complicated by secondary reactions which it will be always necessary to take into account when it is desired to extend the method to other separations than that of nickel and zinc.

1. The concentration of the zinc sulphate should not be too great, for then this salt on the one hand and the dilute

\* The idea of solution-pressure has been suggested in the ionic theory by the analogy which has been established between the tendency which bodies have of becoming ionised, and the tendency which bodies have of passing into the state of vapour. Just as the measure of the second is called the vapour-pressure, so the first has been called the solution-pressure.

† The order of the solution-pressures is the same as that of the polarisation potentials.

‡ In the Nernst formula we have put  $C$  and  $C_1$  as the concentration of the metals, in place of their osmotic pressures  $p$  and  $p_1$ , which should have been used, because  $C$  and  $C_1$  are proportional to  $p$  and  $p_1$  on account of the relatively great dilution of the solutions we have employed.

solution of zinc sulphate which is formed on the other hand by the solution of the zinc in the inner cell, will form a concentration cell ( $Zn | ZnSO_4 \text{ dilute} || Pt | ZnSO_4 \text{ concentrated}; Zn \text{ and } Pt \text{ representing the poles of the cell, that is, the zinc disc and the platinum electrode}$ ). Otherwise the total E.M.F. of the system may reach such a point that the zinc can be precipitated with the nickel, which we found to be the case when the nickel was accompanied by 2 grms. and more of zinc. That is why we have recommended that there should not be more than 1 grm. of zinc in the nickel solution.

2. The sulphate of zinc formed in the solution of magnesium sulphate by the solution of the zinc, attacks the disc of zinc, especially when warm; hence a voltaic couple which produces a new increase of the E.M.F.; an increase which we have endeavoured to diminish as much as possible by arranging the cell so that the disc is in the coolest part of the apparatus. The concentration of zinc sulphate increases as the nickel is deposited, causing the attack on the zinc disc and the E.M.F. to become greater; this would end by favouring the deposit of zinc with the nickel. Fortunately this concentration decreases the E.M.F. of the concentration element spoken of in the previous paragraph, as well as the E.M.F. of the principal element (see Nernst formula), so that one can easily deposit 0.45 grm. of pure nickel even when accompanied by 1 grm. of zinc. When nickel alone is present, one can deposit still more, for in this case there is no concentration element. Hence it follows that the quantity of pure nickel that can be deposited is greater as the accompanying zinc is less (for then the effect of the concentration element is reduced), and inversely that the quantity of zinc accompanying the nickel can be greater according as the nickel is present in smaller amount (for then the E.M.F. of the cell is reduced). By not having more than 0.45 grm. of nickel and 1 grm. of zinc in the solution, one is always certain to have pure nickel deposited.

3. Finally, there is another secondary phenomenon which does not occur with nickel sulphate, but which is manifest with certain other salts. This can be discussed for the sake of completeness. We mean the migration of the salts in the course of the electrolysis from the cathode (here the platinum electrode) to the anode (here the precipitating metal). This is Hittorf's phenomenon. For each metal, then, it is necessary to find out if it forms a salt which does not show the Hittorf phenomenon, and if it is preferable to work at one temperature more than another, for the losses in concentration round the cathode (and around the anode) vary with the temperature. The method cannot be applied to every class of salt, and so is not general. It cannot be used for copper in particular, the  $Cu^{++}$  ions passing very rapidly to the anode cell.

(To be continued).

## ESTIMATION OF PERSULPHATES.

By C. MARIE and L. J. BUNEL.

THE methods in use up to the present for the estimation of persulphates can be divided into two groups:—

1. Those which are based on the oxidising power of the salt.
2. Those which depend on the estimation of the acid liberated during their decomposition.

Le Blanc and Eckardt, Mondolfo, Grutzner, and others have all proposed various methods; while Peters and Moody, who have made comparative tests of them all, do not consider the iodometric methods of Mondolfo and Namias to be exact, but they recommend the methods of Le Blanc and Grutzner.

More recently Taruggi has proposed the acidimetric estimation of the sulphuric acid set free during the decomposition of the persulphate in the reaction:—



The author uses phthalein for the titration, and considers that a total decomposition may be effected by boiling for twenty minutes.

In the case of persulphate of ammonia, it is necessary to operate in the presence of an amount of titrated soda sufficient to replace the ammonia of the salt. By proceeding differently we obtain erroneous results; we shall refer to this question later on.

Now, in undertaking these experiments with the object of examining the quantitative formation of persulphates by electrolysis, we found that the time of boiling mentioned was insufficient to obtain the complete decomposition of the persulphate.

We prepared a decinormal solution of pure dry persulphate of potassium. The estimation of the potassium on a portion gave for 1 molecule of  $SO_4K$ :— $K = 38.8$  found, 39 theory. Twenty-five c.c. were boiled for fifteen minutes, cooled, and titrated with methyl-orange; the titration being made in the cold, the decomposition of the salt does not continue.

The neutralised solution is again boiled; every five minutes it is cooled and titrated; this is repeated for forty minutes. The results are given in the following table:—

| $SO_4K$ N/10. | Time of boiling. | $NaOH$ N/10. |
|---------------|------------------|--------------|
| C.c.          | Mins.            |              |
| 25            | 15               | 18.5         |
| 25            | 20               | 23.9         |
| 25            | 25               | 24.6         |
| 25            | 30               | 24.8         |
| 25            | 35               | 25.05        |
| 25            | 40               | 25.05        |

The experiments repeated with sodic and ammoniac persulphates gave the same results; thus, a minimal boiling of thirty-five minutes is necessary to bring about the complete decomposition of an alkaline persulphate in aqueous solution. In this manner we have been induced to examine the action of a large number of bodies with the object of finding one which would accelerate the decomposition of the persulphate; methylic alcohol has given us the best results.

If we treat a solution of a persulphate with methylic alcohol in excess and then boil, the persulphate transforms a portion of the alcohol into aldehyde, according to a well known reaction. We have made quite certain that no formic acid is formed.

We have tested this method with the pure persulphates of potassium and ammonium; compared with that of Le Blanc and Eckhardt, this method has given the most concordant results. The results are given in the following table:—

| $SO_4K$ N/10.    | Alcohol. | Time of boiling. | $NaOH$ N/10. |
|------------------|----------|------------------|--------------|
| C.c.             |          | Mins.            |              |
| 25               | 2        | 15               | 24.9—25.05   |
| 25               | 2        | 25               | 24.9—25.05   |
| 25               | 20       | 15               | 24.95        |
| $SO_4NH_4$ N/10. |          |                  |              |
| 25               | 2        | 15               | 24.9—25.0    |
| 25               | 20       | 25               | 25.0         |

By this method we have titrated impure persulphate of soda and found:—

|                           | By our method. | By Le Blanc's method. |
|---------------------------|----------------|-----------------------|
| $SO_4Na$ , per cent . . . | 87.5           | 87.4                  |

These results justify us in recommending the following procedure for the titration of alkaline persulphates:—

Dissolve about 0.3 to 0.4 grm. of the sample in 100 c.c. of water; neutralise the solution, which is generally acid, in the presence of methyl-orange; then add 2 c.c. of methylic alcohol, heat for five minutes to 70–80°, and then boil for ten minutes, cool, and titrate with methylic alcohol and decinormal soda.

|                                                              |  |
|--------------------------------------------------------------|--|
| 1 c.c. of decinormal soda corresponds to 0.0135 grm. $SO_4K$ |  |
| 1 " " " " 0.0119 " $SO_4Na$ .                                |  |
| 1 " " " " 0.0114 " $SO_4Am$ .                                |  |

We have already seen that in the titration of persulphate of ammonia, M. Taruggi recommends boiling the solution treated with a sufficient quantity of titrated soda to completely displace the ammonia in the salt.

Without taking this precaution we obtain results much too high; for example:—

| SO <sub>4</sub> NH <sub>4</sub> N/10. | Time of boiling. | NaOH N/10.       |
|---------------------------------------|------------------|------------------|
| C.c.                                  |                  |                  |
| 25                                    | 15 mins.         | 26.8 (theory 25) |
| 25                                    | 20 "             | 27.6             |
| 25                                    | 1 hour           | 28.85            |

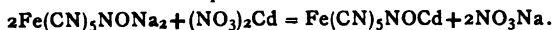
This does not occur in the presence of methylic alcohol. We think it is correct to attribute this fact to the oxidation of the ammonia, which is transformed into nitrogen and water by the oxygen of the persulphate.

In fact, if we boil a solution of persulphate of ammonia in a vessel traversed by a current of carbonic acid, and collect the gases given off in a solution of potash, we find a considerable residue of nitrogen after absorbing the oxygen with an alkaline pyrogallate. If this phenomenon is not produced in the presence of methylic alcohol, it is because the speed of oxidation of the ammonia is very slow compared with that of the alcohol. This, however, requires further study, which we propose to carry out.—*Bull. Soc. Chim.*, Series 3, vol., xxix., No. 18.

#### VOLUMETRIC ESTIMATION OF THE ALKALINE NITROPRUSSATES, AND OF THE SOLUBLE SALTS OF CADMIUM.

By MM. FONZES-DIACON and CARQUET.

THE volumetric estimation of a soluble nitroprussiate is based on the following principle:—Nitroprussiate of cadmium being insoluble in water, if to a solution of a nitroprussiate we add a known volume of a titrated solution of nitrate of cadmium, a precipitate will be formed containing the whole of the nitroprussiate:—



Thus by estimating volumetrically the excess of nitrate of cadmium, we can determine the weight of the corresponding nitroprussiate; but we might also estimate this latter compound directly by the volumetric analysis of the precipitated nitroprussiate of cadmium.

These two methods will serve as a check on each other, and we can take the mean of the two observations.

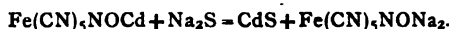
1. *Estimation of the Excess of Nitrate of Cadmium.*—We first prepare a titrated solution of this salt by dissolving 8 or 10 grms. in a litre of water, and determining the value volumetrically by means of a titrated solution of sulphide of sodium, using a few drops of nitroprussiate of soda as an indicator,  $(\text{NO}_3)_2\text{Cd} + \text{SNa}_2 = \text{SCd} + 2\text{NO}_3\text{Na}$ .

At first a yellow precipitate of sulphide of cadmium is formed, and the end of the reaction is shown by the appearance of the violet colouration caused by the action of a slight excess of sulphide on the nitroprussiate of soda.

We then add to a known volume of the nitroprussiate solution under examination a known excess volume of the titrated solution of nitrate of cadmium; the precipitate is separated by filtration and washed, and in the filtrate mixed with the washings we estimate volumetrically the excess of nitrate of cadmium; for this purpose we add a few drops of nitroprussiate, and, by means of a burette, add the titrated solution of sulphide of sodium until a violet colour appears.

2. *Analysis of the Nitroprussiate of Cadmium.*—The precipitate collected on the filter is dissolved in a little ammoniacal water; into this solution we pour, drop by drop, a titrated solution of sulphide of sodium; there is first formed a yellow precipitate of sulphide of cadmium, and at the end of the operation there appears the violet colour-

ation caused by a slight excess of sulphide on the nitroprussiate of soda formed:—



The verification of the sensitiveness of this method has shown us that it has all the accuracy required of a volumetric method.

It is of general application, except always in the presence of cyanides and of ferro- and ferri-cyanides; we can, however, get rid of the former by passing a current of carbonic acid gas through the boiling solution; this drives off the hydrocyanic acid, and we can then precipitate the latter salts by the addition of sulphate of zinc. In the filtered solution we can estimate the nitroprussiate by the above mentioned method.

This method has enabled us to detect and estimate nitroprussiate of soda in the organism during a series of experiments undertaken to determine the toxicity of this salt.

Thus, this method is applicable also to the volumetric estimation of a soluble salt of cadmium; for this purpose we use a titrated solution of sulphide of sodium in the presence of nitroprussiate of sodium, which serves as an indicator.—*Bull. Soc. Chim.*, Series 3, vol. xix., No. 13.

## PROCEEDINGS OF SOCIETIES.

### CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, February 17th, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MR. E. R. BUGGÉ was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. William Barbour, M.A., B.Sc., Grove Villa, Waltham Cross; R. H. Durward Benn, Westmount, Montreal, Canada; Ellis Clayton, 6, Spring Hurst Road, Saltire; Percy Kent Le May, 6, Lothair Villas, Hatfield, Herts; Franklin E. Robertson, Harders Road, Peckham, S.E.; Thomas G. Shacklady, Addiscombe Villas, Cliffe-at-Hoo, Kent.

It was announced that the following changes in the Officers and Council were proposed by the Council:—

As Treasurer: Dr. A. Scott, F.R.S., *vice* Dr. H. T. Brown, F.R.S.

As Secretary: Dr. M. O. Forster, *vice* Dr. A. Scott, F.R.S.

As Vice-Presidents: Dr. H. T. Brown, F.R.S., and Prof. H. B. Dixon, F.R.S., *vice* Prof. H. McLeod, F.R.S., and Prof. H. A. Miers, F.R.S.

As Ordinary Members of Council: Dr. Bernard Dyer, Mr. A. D. Hall, Dr. A. Lapworth, Prof. J. M. Thomson, F.R.S., *vice* Dr. M. O. Forster, Mr. S. U. Pickering, F.R.S., Dr. J. A. Voelcker, and Prof. J. Walker, F.R.S.

Dr. L. T. Thorne, Dr. G. T. Moody, and Dr. J. Wade were elected to audit the Society's accounts.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—John Prest Ackroyd, B.Sc.; Allen Baguley, B.Sc.; Charles Thomas Bennett; Alick Cole Benson; George Edward P. Broderick, B.Sc.; Alfred Denys Cowper; Julien Drugman, Ph.D.; Nevil Norton Evans; James Rocheid Forrest; George Fry; Robert Gawler, M.Sc.; Anu Ghose; Harry James Glover; John Augustus Goodson; John Monteath Guthrie; Leo Frank Guttmann, Ph.D.; Alfred Henry Hoit; Cyril Douglas McCourt; Francis D'Oyley Mears, jun.; Bernard Middleditch, B.A.; Jack Percival Montgomery; Benjamin L. Murray, B.Sc.; Thomas S. Patterson, Ph.D.; Bertram Prentice, Ph.D., D.Sc.; Jatindranath Sen, M.A.; Alfred Shrubsole; Raymond Louis Siau; Samuel John Smith; Henry Ernest Stevenson; Frederick Henry Streatfeild; Charles Edward

Thompson, Hubert Thompson, B.Sc.; Alfred Tingle, B.Sc.; Ph.D.; William Wood Underhill; Léon Edward Welling; Francis Langston Watt; Charles Edward Whiteley; William Henry Woodcock.

Of the following papers, those marked \* were read:—

\*24. "Observations on some Intramolecular and Originally Reversible Changes Extending over Prolonged Periods of Time." By RICHARD JOHN FRISWELL.

The question of the limitation of chemical action to a certain range of temperature was discussed in the light of W. R. Grove's experiments on the dissociation of water by heat, described in the Bakerian Lecture (*Phil. Trans.*, 1847, cxxvii., 1), and of recent experiments at very low temperatures, and it was pointed out that, as a rule, reactions are hastened by elevation of temperature, and hence their course has been less studied than their products.

Reference was made to V. Meyer's labile hydrogen atom, and some results obtained by the author and A. G. Green (*Trans.*, 1885, xlvii., 917; 1886, xlix., 746) were discussed. It is suggested (1) that the labile condition is not confined to hydrogen; (2) that the constitution of a compound is only relative, and exists only so long as the substance is submitted to a certain definite stress; (3) that different stresses develop dissimilar constitutions, and that therefore the constitution of a compound depends on its environment.

Experiments on the changes occurring during very long periods of time were described, these consisting in the slow appropriation by aminoazobenzene base from a solution of aniline hydrochloride of sufficient hydrochloric acid to saturate itself; this reaction was shown to occur even in the presence of much free aniline, and the variations due to changes of temperature were described.

These and analogous experiments are held to justify the arguments as to the effects of stress on the successive disruption of the molecules of aniline hydrochloride and aminoazobenzene hydrochloride.

Preparations illustrating the above-described experiments were exhibited.

#### DISCUSSION.

Dr. MORGAN suggested that the interaction occurring between aniline hydrochloride and aminoazobenzene might be explained in terms of the ordinary theories of chemical equilibrium.

Aniline hydrochloride, in aqueous solution, undergoes dissociation, and attains a state of equilibrium, which may be represented approximately by the equation  $C_6H_5NH_3Cl \rightleftharpoons C_6H_5NH_2 + HCl$ . This equilibrium is disturbed by the introduction of aminoazobenzene, because this base forms a sparingly soluble hydrochloride, and thus gradually abstracts the free acid from the solution. Excess of aniline hinders this change owing to the fact that, being itself a product of the dissociation of the soluble hydrochloride, it reverses the foregoing balanced reaction, in accordance with the law of mass action, thereby decreasing the amount of available hydrochloric acid.

Dr. HEWITT asked whether the proof of the existence of a diazonium salt in an acid solution of diazoaminobenzene during the period of isomerisation to aminoazobenzene did not depend on the coupling properties of such a solution; if so, the demonstration is not conclusive, since it has been known for years that diazoamino-compounds couple with phenols even in the absence of acids. One of the earliest known cases of this reaction was discovered by Heumann and Economides, who obtained benzeneazophenol from diazoaminobenzene and phenol (*Ber.*, 1887, xx., 372).

In reply to Dr. Hewitt, Mr. FRISWELL said that a full account of the evidence for the presence of diazobenzene chloride in the solution during the conversion of the diazoaminobenzene into aminoazobenzene would be found in the papers published in conjunction with Prof. A. G. Green (*loc. cit.*). Shortly, it consisted in treating the separated solution with phenols and amines, whereby known azo-dyes were produced and identified.

In reply to Dr. Morgan, he had no objection to make to

the explanation based on the suggested tendency to dissociation of aniline hydrochloride in aqueous solution. He had endeavoured to overcome this tendency by the addition of free aniline, but this only delayed the abstraction of the acid by the aminoazo-base, and, in his opinion, furnished additional evidence in favour of the stress hypothesis which he had advocated.

\*25. "Note on a Magnesium Oxybromide." By GEORGE WILLIAM FRASER HOLROYD.

An ethereal solution of magnesium phenyl bromide, obtained by Grignard's method (*Ann. Chim. Phys.*, 1901, xxiv., 437), when saturated with acetylene and left in a stoppered vessel for several days, deposited clear, colourless crystals having the form either of octohedra or of combinations of the octohedron and cube. The yield of crystals was about 4 grms. from 50 grms. of bromobenzene.

The volume of the solution before passing the acetylene was 220 c.c., and 52 grms. of bromobenzene were employed. The acetylene, dried with phosphorus pentoxide, was passed through successive portions of 30 c.c. of this solution, the current of gas being continued for about four hours, and the flasks being kept for two or three days or longer before removing the crystals which are gradually deposited. The ethereal solution diminished to about two-thirds of its original volume during the saturation with acetylene.

The crystals, which vary considerably in size when obtained from distinct preparations, are hygroscopic and very soft; they were not re-crystallised for analysis, but rapidly dried on filter-paper, scraped with a sharp nickel spatula, and analysed as quickly as possible.

0.1037 gave 0.1291 AgBr. Br = 53.00.

0.0804 " 0.048 MgSO<sub>4</sub>. Mg = 10.86.

C<sub>8</sub>H<sub>21</sub>O<sub>3</sub>Br<sub>3</sub>Mg<sub>2</sub> requires Br = 52.86; Mg = 10.49 per cent.

The substance is decomposed by water, heat being generated during the reaction; magnesium hydroxide is precipitated, and ether is set free.

The ether was estimated by introducing a crystal into a eudiometer standing over mercury; dry oxygen and two drops of water were then added successively, the apparatus was surrounded by a steam jacket and an electric spark passed through the gaseous mixture. The carbon dioxide produced was measured by caustic potash absorption, after decomposing any magnesium carbonate present by the addition of three drops of dilute sulphuric acid.

0.0221 gave 8.44 c.c. CO<sub>2</sub>; Mg<sub>2</sub>Br<sub>3</sub>C<sub>8</sub>H<sub>21</sub>O<sub>3</sub> requires 8.73 c.c. CO<sub>2</sub>.

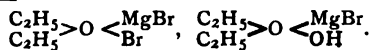
Oxygen used = 13.34 c.c.; Mg<sub>2</sub>Br<sub>3</sub>C<sub>8</sub>H<sub>21</sub>O<sub>3</sub> requires 13.1 c.c. oxygen.

The analyses point to the formula Mg<sub>2</sub>Br<sub>3</sub>·OH, 2(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O, and the substance doubtless owes its origin to the action of small quantities of water on the magnesium phenyl bromide, C<sub>6</sub>H<sub>5</sub>MgBr + H<sub>2</sub>O = C<sub>6</sub>H<sub>6</sub> + MgBr·OH, the magnesium oxybromide then combining with ether and magnesium bromide; the latter is always a by-product of the action of bromobenzene on magnesium in ethereal solution, the acetylene serving merely to evaporate the ether.

Zelinsky (*Chem. Centr.*, 1903, ii., 277), by the interaction of magnesium, iodine, and ether, obtained a compound to which he assigns the formula—



and the compounds MgBr<sub>2</sub>·3(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O and MgBr<sub>2</sub>·(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O have also been produced. Zelinsky considers that these compounds play the rôle of catalysts in the formation of the mixed organo-magnesium compounds. The compound obtained by the author is evidently related to these products, and should perhaps be represented by the formula—



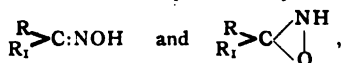
\*26. "The Arrangement in Space of the Groups combined with the Tervalent Nitrogen Atom." By FREDERIC STANLEY KIPPING and ARTHUR HENRY SALWAY.

The Hantzsch and Werner hypothesis regarding the tervalent nitrogen atom indicates the existence of enantiomorphously related isomerides in compounds of the type  $\text{NR}_1\text{R}_2\text{R}_3$ , and one of the objects of this investigation was either to isolate, if possible, such isomerides or to demonstrate their non-existence.

Since an externally compensated acid chloride may be used for the detection of asymmetry in bases where the asymmetry is due to a carbon atom (Kipping and Hall, *Trans.*, 1901, lxxix., 444), it seemed probable that, if tervalent nitrogen compounds were asymmetrical, their asymmetry could be detected in like manner, but no evidence of the existence of isomerides was obtained on examining the products of the interaction of *dl*-benzylmethylacetyl chloride with methylaniline, *p*-toluidine, benzyaniline, and phenylhydrazine. *p*-Toluidine and benzyaniline also gave negative results with optically active benzylmethylacetyl chloride. Although the introduction of one centre of asymmetry failed to afford separable isomerides, it seemed highly probable that the introduction of a second centre of asymmetry would give a satisfactory result if enantiomorphously related derivatives of tervalent nitrogen were really capable of existence. The products from *d*-benzylmethylacetyl chloride and *l*-methylamine, *d*-hydrindamine, *l*-methylhydrindamine, and *l*-phenylethylamine, however, retained their uniform character after fractional crystallisation. These results indicate that the three radicles, together with the tervalent nitrogen itself, are situated in one plane; that two of the radicles are symmetrically arranged with respect to the third, and that in all probability this is true of any two; that is to say, the whole arrangement is the most symmetrical one possible.

The substituted amides derived from the interaction of benzylmethylacetyl chloride and the foregoing inactive and active bases were described.

Assuming that the isomerism of the syn- and anti-forms of oximes is structural and represented by the formulæ:—



the latter structure, since it contains an asymmetric carbon atom, should exist in enantiomorphously related forms, and the introduction of an optically active acid chloride should produce two non-enantiomorphously related separable isomerides. The action of benzylmethylacetyl chloride on benzoinoxime was studied, but instead of the desired acyl derivative, the following products were obtained:—Benzil, benzoin, benzaldehyde, benzonitrile, ammonium chloride, hydrogen chloride, and benzylmethylacetic acid, together with a compound melting at  $126^\circ$ . Attempts to obtain a benzylmethylacetyl derivative from  $\alpha$ -benzaldoxime were also unsuccessful, decomposition taking place with the formation of benzonitrile.

Benzoyl chloride interacts normally with benzoinoxime, giving *benzoinoxime benzoate*,  $\text{CHPh}(\text{OH})\cdot\text{CPh}\cdot\text{NOBz}$ , which crystallises from alcohol in leaf-like plates melting at  $165$ – $166^\circ$ .

*Benzoylbenzoinoxime*,  $\text{CHPh}(\text{OBz})\text{CPh}\cdot\text{NOH}$ , prepared by treating benzoylbenzoin with hydroxylamine, crystallises from alcohol in globular clusters melting at  $148^\circ$ .

*dl*-Benzylmethylacetyl chloride gives, with *dl*-hydrindamine two isomeric *dl*-benzylmethylacetohydrindamides, which can be easily separated; these two compounds melt at  $110$ – $111^\circ$  and  $119$ – $120^\circ$  respectively, and their formation affords proof of the asymmetry of the hydrindamine molecule.

*d*-Benzylmethylacetyl chloride and *dl*-hydrindamine give a mixture of the isomeric amides *dAdB* and *dAlB*, from which the derivative of the *d*-base is easily isolated; the enantiomorphously related components of *dl*- $\alpha$ -phenylethylamine may be separated in a similar manner, the com-

pound,  $\text{CH}_2\text{Ph}\cdot\text{CHMe}\cdot\text{CO}\cdot\text{NH}\cdot\text{CHMe}\cdot\text{Ph}$ , of the *l*-base being easily isolated.

27. "The Esterification of *r*-Mandelic Acid by Menthol and Borneol." By ALEXANDER MCKENZIE.

The results recorded in this research deal with the method of resolving *i*-compounds devised by Marckwald and the author (*Ber.*, 1899, xxxii., 2130; 1900, xxxiii., 208; 1901, xxxiv., 469. Compare Walden, *Ber.*, 1899, xxxii., 2703; E. Fischer, *Ber.*, 1899, xxxii., 3617).

When *r*-mandelic acid was heated with *l*-borneol, the unesterified acid was laevorotatory, whilst the mixture of esters yielded a laevorotatory acid. When *l*-bornyl *dl*-mandelate was submitted to fractional hydrolysis, a laevorotatory acid was obtained from the initial hydrolysis, and an inactive acid from the final hydrolysis of the residual esters by an excess of alcoholic potassium hydroxide. *d*-Mandelic acid was isolated from the dextrorotatory acid obtained from the initial hydrolysis of *l*-menthyl *dl*-mandelate. *l*-Menthyl *dl*-mandelate can yield either a dextrorotatory or a laevorotatory acid from the initial fractional hydrolysis according to the amount of alkali used. *l*-Menthyl *dl*-mandelate boils at  $225^\circ$  under 30 m.m. pressure, and melts at  $85$ – $86^\circ$ ; it has  $[\alpha]_D^{18} -74.2^\circ$  ( $c=10.890$ ) in ethyl-alcoholic solution; it is partially racemic, and is not resolved into *l*-menthyl *d*-mandelate and *l*-menthyl *l*-mandelate when repeatedly crystallised from light petroleum at the temperature of the laboratory. Potassium *l*-mandelate is completely racemised when heated with a large excess of potassium hydroxide in aqueous or ethyl-alcoholic solution.

28. "Certain Organic Phosphorus Compounds." By AUGUSTUS EDWARD DIXON.

In pursuing the study of the interaction of phosphorus halides with metallic thiocyanates (*Trans.*, 1901, lxxix., 547), the author has now succeeded in isolating phosphorus and phosphoryl "thiocyanates" respectively.

Phosphorus trithiocyanate,  $\text{P}(\text{CNS})_3$ , obtained from phosphorus trichloride and dry ammonium thiocyanate in presence of benzene, is an almost colourless oil boiling at  $163^\circ$  under 15 m.m. pressure, and having a sp. gr. 1.487 at  $15.5^\circ$ . When treated with water, a portion was quickly hydrolysed, forming phosphorous and thiocyanic acids; the remainder was hydrolysed exceedingly slowly, but otherwise no material difference in properties or composition was observed between this residue and a distillate not subjected to the action of water.

Phosphoryl trithiocyanate,  $\text{PO}(\text{CNS})_3$ , obtained from phosphorus oxychloride, is a clear, pale yellow, highly refractive oil, boiling at  $175^\circ$  under 21 m.m. pressure, and completely hydrolysed by cold water into phosphoric and thiocyanic acids, together with some isoperthiocyanic acid, resulting from the interaction of these products; it has a sp. gr. 1.520 at  $13.5^\circ$ .

These substances not only behave as thiocyanates, but also, to some extent, manifest the properties of thiocarbimides, being freely desulphurised by alkaline salts of lead and silver; they unite spontaneously with aniline, &c., fixing either one or three molecular proportions, according to the amount presented, to form amorphous solids, insoluble in cold water. The additive products are readily hydrolysed by contact with hot water, yielding in all cases approximately one molecular proportion of monosubstituted thiourea; a little hydrogen sulphide is evolved, but otherwise the rest of the contained sulphur appears as thiocyanic acid.

Certain tests which have been employed to distinguish between thiocyanates and thiocarbimides (such as treatment of the alcoholic solution with sodium, or the action of thioacetic acid) cannot be safely accepted as final where acyl thiocyanates are concerned, all the latter so far examined being tautomeric in the sense that, under suitable conditions, they can behave as thiocarbimides to a greater or less extent if their power in this direction is measured by their capacity to fix aniline in the form of an immediate derivative of normal phenylthiocarbamide,



The study of these supposed tautomeric phenomena is being continued.

29. "Note on the Relation between the Chemical Composition of some Organic Substances and the Density of their Solutions." By CHARLES EDWARD FAWSITT.

The density of aqueous solutions of different salts in equivalent quantities is an additive property, and the principal relations are given by Valson's Law of Moduli. The relation of density in solutions of organic compounds (feeble electrolytes or non-electrolytes) to chemical composition or constitution is not yet so fully understood.

The author, in continuing an investigation on the amides (*Proc. Roy. Soc. Edin.*, 1904, xxv., 51), has measured the density of aqueous solutions of a number of amides at 25°, when the solutions contained the grm.-molecular weight of the substance dissolved in one litre. The densities of urea, methylurea, and *as*-dimethylurea solutions are 1.0155, 1.0137, and 1.0107 respectively; a comparison of these numbers with those obtained by Kanitz (*Zeit. Physik. Chem.*, 1897, xxii., 336) for ammonia, methylamine, and dimethylamine, namely, 0.9932, 0.9886, and 0.9856, shows that the differences for the corresponding numbers are fairly close to one another.

The densities of solutions of acetamide, propionamide, and butyramide are 1.0040, 1.0028, and 1.0006.

The values obtained for acetic, propionic, and butyric acids by Reyhes (*Zeit. Physik. Chem.*, 1888, ii., 744) are 1.0084, 1.0066, and 1.0040, and show a similar relationship.

From these results, it will be seen that the density of such solutions is to a great extent an additive property, but constitution also plays some part, because isomeric substances give different values.

30. "The so-called 'Hydrocellulose.'" By ARTHUR LANDAUER STERN.

When cellulose is exposed to the action of dilute acids under certain conditions, the tenacity of the fibres is destroyed, and it falls to a powder which has been called hydrocellulose, and stated to have the empirical formula  $C_{12}H_{22}O_{11}$ .

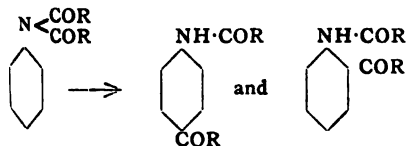
It is now shown that when the above reaction takes place, instead of a gain in weight, as theory indicates, there is invariably a loss, and that a small amount of soluble matter is formed, a portion of which, in all probability, is *d*-glucose.

The elementary composition of the powder is also shown to be identical with that of cellulose, the previous statements bearing on this point being founded on faulty experimental methods.

A hydrated cellulose is not formed under these conditions, but a hydrolysis takes place similar to that undergone by other carbohydrates under comparable conditions.

31. "Isomeric Change of Diacylanilides into Acylamino-ketones." By FREDERICK DANIEL CHATTAWAY.

When the diacylanilides are heated in presence of hydrogen chloride or zinc chloride, intramolecular rearrangement takes place and the isomeric acylamino-ketones are produced thus:—



Such isomeric changes as have been studied follow an exactly similar course to that of other analogous transformations in which atoms or groups of atoms pass from the nitrogen into an ortho- or para-position in the ring.

The introduction of a second acyl group into the nucleus has, however, not yet been effected (compare *Proc.*, 1902, xviii., 173; 1903, xix., 50, 57, 106, 124).

32. "Intramolecular Re-arrangement in Derivatives of the Aromatic Aminoketones." By FREDERICK DANIEL CHATTAWAY.

The acylchloroamino-derivatives of the aromatic ketones readily undergo the intramolecular re-arrangement characteristic of substituted aromatic chloroamines in which the halogen linked to the nitrogen changes place with a hydrogen atom attached to the ring in an ortho- or para position. The conditions necessary for the transformations to take place were indicated, and a series of new aromatic chloroaminoketones with their acyl derivatives was described.

#### Annual General Meeting.

The Annual General Meeting of the Society for the Election of Officers and other business will be held on Wednesday, March 23rd, at 5.30 p.m.

#### THE FARADAY SOCIETY.

Ordinary Meeting, February 2nd, 1904.

Dr. J. W. SWAN, F.R.S., President, in the Chair.

THE PRESIDENT referred in terms of deep sympathy to the recent lamented death of Mr. W. G. McMillan, and moved that resolutions giving expression to their sense of loss of a valued friend and colleague be sent to Mrs. McMillan and to the Council of the Institution of Electrical Engineers.

MR. JONAS read an abstract of a paper by Mr. S. O. COWPER-COLES, entitled "Notes on the Welding of Aluminium."

The author commenced his paper by stating that soldered aluminium joints have proved unsatisfactory, as they will not stand the test of time, because galvanic action takes place between the aluminium and solder. One of the chief difficulties encountered other than the formation of oxide in soldering aluminium is, that a few degrees below its welding-point it passes into a pasty or brittle state, and being a very good conductor of heat, the solder very rapidly cools and freezes before it has time to flow sufficiently. He then proceeded to describe Dick's machine for welding aluminium by the removal of the oxide mechanically, combined with pressure. Reference was also made to Heraeus's process of welding aluminium, which consists in heating the aluminium in a reducing atmosphere until it reaches the pasty stage, when the joint is made by kneading and hammering. Emmé's process, which is somewhat similar, consists in heating the aluminium up to 600° C., and welding by hammering. The electric welding of aluminium has not proved commercially successful. A description was given of Schmidt's electric welding apparatus, which consists of a carbon stick, which is used in much the same way as a soldering iron, by being moved over the portion to be welded to remove the oxide, the aluminium forming one electrode, the carbon stick the other electrode. Jones's machine for welding aluminium tubes was also illustrated; a strip of aluminium is wrapped round a mandrel, and the joints are fixed together by passing a low tension current through the portions to be welded. The author then went on to describe his own process, which requires no flux or solder, and does not necessitate the hammering of the joint when in the pasty state, the process being especially suitable for wire, rods, and tubes. The machine employed is fitted with clamping screws which are controlled by levers. The aluminium is heated by a benzine lamp, and when raised to the point of fusion, pressure is applied to the levers, which causes the aluminium to be welded to unite, a ring of metal being squeezed out. This ring, which is largely composed of oxide, acts as an insulating and supporting collar, the molten metal being retained within it. Water is then projected on to the joint by turning a handle, at the same time a screen is moved in front of the flame. The rod is then removed, and when

the collar is filed off the joint will be found to be as strong as the rest of the metal. A table was given showing the tensile strength of twelve consecutive welds; in all the tests the specimen broke at some distance from the weld, and in no case did it actually break through the weld. Illustrations were given demonstrating how molten aluminium can be encased in a shell of aluminium oxide.

Mr. D. A. SUTHERLAND said the principle of the method was familiar to metallurgists who had to report on these matters. The great difficulty was where large pieces of aluminium had to be welded.

Mr. MURRAY MORRISON agreed with the last speaker; he himself had welded many joints in this way, although he did not quench. He always found that pressure was necessary. The chief novelty of the Cowper-Coles process lay in the apparatus.

Mr. BLOUNT said when drawn wires were welded it was essential that the joint be drawn or worked in some way, otherwise being cast metal it would be weaker than the rest of the wire.

Prof. HUNTINGTON thought that the chief novelty in the process lay in the fact that the metal was actually fused.

Mr. E. KILBURN SCOTT said that the important point to remember was the value of the hard outer skin of the metal. If aluminium wires were used for transmission of electricity, uniformity of section was unimportant, and the blob at the weld need not be removed. But otherwise, *e.g.*, if used as trolley wires, it would be necessary to harden the joint in some way; this might be done by burnishing with agate. He showed by a diagram how a connection might be made without welding.

The PRESIDENT said that the apparatus they would shortly see at work showed a thorough appreciation of the conditions necessary to effect a weld, and in dealing with the film of oxide so as to hold together the molten metal made a virtue of a vice.

Mr. JONAS, in his reply, drew attention to the tests of tensile strengths of jointed rods given in the paper. In reply to Mr. Blount, the joints proved to be the strongest part of the rods. He would remark that the quenching was an essential feature of the process, and one which distinguished it from all others, the process being primarily a fusion process.

Dr. F. M. PERKIN then read an abstract of a paper by M. HOLLARD, entitled "*Some Applications of the Theory of Electrolysis to the Separation of Metals from one Another.*" (See p. 110).

Mr. BLOUNT remarked that M. Hollard's method of preparing the cathode by throwing down on it the metal that is to be deposited, is one of extreme interest. The accuracy of the first method described was not sufficiently high for ordinary analytical purposes.

Dr. T. M. LOWRY, referring to the backward migration of the ions, said that as a rule this is much more strongly marked with a membrane than with a porous pot partition. Hittorf had recently been again working on this very subject.

Mr. G. WATSON GRAY read a short preliminary note describing an explosion of some high grade ferro-silicon which occurred spontaneously a short time ago at Liverpool.

The gases evolved on boiling a specimen in distilled water were found to contain  $\text{PH}_3$  and  $\text{AsH}_3$ . The former was in the greater proportion, and to that probably the explosions—which had created a considerable stir in Liverpool and which the author had not met with before—were due.

Mr. D. A. SUTHERLAND thought that the presence of only 0.1 per cent phosphorus made it very difficult to account for the explosions being due to phosphoretted hydrogen. It was essential to know the previous history of the metal.

Mr. R. H. HARLAND, who had gone into the matter, believed the explosion was due to calcium carbide, which had been previously made in the same furnace.

Mr. E. KILBURN SCOTT agreed with the last speaker; he had had a similar experience in making magnesite in furnaces that had been used for calcium carbide.

Mr. MURRAY MORRISON observed that all the ingredients necessary for  $\text{CaC}_2$  are present in the manufacture of ferro-silicon.

Prof. HUNTINGTON said that ordinary ferro-manganese of over 80 per cent made in the blast-furnace disintegrates. This may be an action of the same kind; it is only a matter of degree.

THE PRESIDENT said there was obviously a difference of opinion as regards the cause of these remarkable explosions; we must therefore wait the results of further inquiries, which he hoped would be presented to the Society, before we could be sure of the true explanation.

Mr. GRAY, in his reply, said there was certainly less than 0.1 per cent of calcium in this particular specimen of ferro-silicon. There was no doubt as to the presence of phosphoretted hydrogen in the gases evolved. He would report further in the matter, and Mr. Harland promised to do the same.

## CORRESPONDENCE.

### WHY ARE SOME ELEMENTS MORE ABUNDANT THAN OTHERS?

*To the Editor of the Chemical News,*

SIR,—In reply to my letter (CHEMICAL NEWS, vol. lxxxix., p. 47), Mr. Kingdon (CHEMICAL NEWS, vol. lxxxix., p. 58) suggests that the greater abundance of certain elements over others is only an apparent effect, because the heavier elements gravitate towards the centre of the earth and the lighter towards the surface; and that the centre of the earth is largely composed of heavy elements, such as gold and platinum.

In writing my former letter I had this possibility in mind (for which reason I wrote "a cause," not "the cause"), but I do not think that this "density" theory gives a full explanation of the facts.

In the first place, there is grave reason to doubt whether the interior of the earth is so very largely composed of very heavy elements. For example, the density of the earth is only 5.5, which is not great, compared with the surface density 2.7, when we consider the tremendous crushing pressure to which the matter in the interior is subjected.

Amagat compressed oxygen gas (3000 atmospheres) until it became denser than water.

Did the interior of the earth consist largely of gold and platinum the density of the earth would be very much greater than 5.5.

The density of the moon is still less than that of the earth; and other celestial bodies possess a still smaller density.

In the second place, there exist very puzzling anomalies as regards the distribution, which the "density" theory does not seem capable of explaining.

For example, why is chlorine (atomic weight 35.5) so much more abundant than F (atomic weight 19), Br (81), or I (126)? Why is argon (40) so much more abundant than neon (20), krypton (81), and xenon (128)? Why are O and Si (16 and 28) so much more abundant than N and P (14 and 32)?

The difference of density cannot account for these cases. Other causes of the different abundance of elements may be—

1. The greater rate of escape into space of the lighter elements, such as H and helium.
2. The different affinities of different elements for heavy metals, which leads to some being stored up in a combined form in greater quantities than others in the interior of the earth.

But even these causes do not account for the facts. For example, the argon group of elements do not unite with

elements, and all are volatile enough to occur in a gaseous form at the surface of the earth. Why then is argon the most abundant? It is neither the lightest nor the heaviest of this family of elements.

Again, bromine and iodine do not possess so great an affinity for other elements as does chlorine (which can displace them from their compounds), and so would not be any more likely than chlorine to be stored up in the interior of the earth.

Moreover, bromides and iodides are unstable under the action of heat—more so than the corresponding chlorides—and so would be even less capable of existing in the interior of the earth than chlorides.

In addition, the metallic iodides and bromides are more volatile and fusible than the corresponding chlorides (*cf.* AgI with AgBr, AgCl), and so would not be expected to occur in the interior of the earth in greater quantity than the chlorides on account of their greater involatility and fusibility.

Finally, the difference in volatility between Br, Cl, and I is so small, and the heat in the interior of the earth so great, that all would be expected to distil out more or less completely from the interior to the surface of the earth.

As a matter of fact, we are acquainted with matter which has come out from depths where the earth is white hot, and we find that Br and I are not more abundant in this matter than chlorine.

It is, in fact, indisputable that chlorine occurs throughout the earth's crust, from depths where it is white hot to the surface, in enormously greater quantities than Br or I.

For this reason I believe that chlorine is not only relatively, but also absolutely, more abundant than Br or I.

Precisely similar considerations apply to S and Se. The selenides are far more unstable under the action of heat than the sulphides. Se has a smaller affinity for metals and elements generally than sulphur, and so could not be expected to be stored up in greater quantities than sulphur at great depths.

Moreover, both elements are volatile enough to readily distil from the hot interior towards the surface of the earth.

Yet sulphur is a far more abundant element in every strata than selenium, even in volcanic lavas which have come out from depths where matter is white hot.

Indeed, I think that all the facts we know point to the conclusion that some elements occur in the universe in far greater quantities than others. Why?

Let us remember—

1. That heavy elements are less abundant than light elements.
2. That the phenomenon of radio-activity shows that heavy elements are less stable than light elements.

Are these two facts unconnected?—I am, &c.,

GEOFFREY MARTIN.

University of Kiel, January 31, 1904.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 5, February 1, 1904.

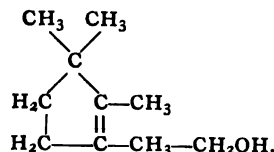
**Action of Carbon on Quicklime at the Temperature of Fusing Platinum.**—Henri Moissan.—The author finds that an intimate mixture of finely-powdered quicklime and sugar-carbon is not attacked when kept for ten minutes at the temperature of fusing platinum. No trace of calcium carbide can be found, and if the tube and its contents are placed in an inverted tube of water, not one bubble of acetylene can be detected even after several days.

**Direct Reduction of Aromatic Halogen Derivatives by Finely-divided Nickel and Hydrogen.**—Paul Sabatier and Alph. Mailhe.—The authors investigate the reaction taking place when reduced nickel in presence of hydrogen causes the direct reduction of the aromatic halogen derivatives without the addition of hydrogen to the radicle.

**Manganous Salts as Oxydases in presence of a Colloid.**—A. Trillat.—The author's experiments show that colloid solutions of manganese obtained in presence of albumin and an alkali possess very remarkable properties. During his researches on the physiological rôle of manganese, M. Bertrand put forward the hypothesis that proteid matter combined with this metal is in the most advantageous form for oxidising, and this opinion is confirmed by the author's results.

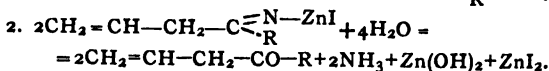
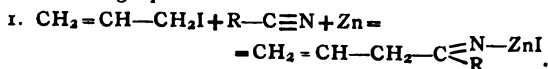
**Mixtures of Antimony Trisulphide and Antimony.**—H. Pélabon.—Antimony trisulphide and antimony, when intimately mixed and raised to a temperature above the fusing-point of antimony, do not usually give a homogeneous liquid, but two superposed liquids. The author investigates the causes of this phenomenon.

**An Isomer of Borneol. Campholenic Alcohol and certain Campholenic Derivatives.**—A. Béhal.—The author prepares campholenic alcohol, starting from inactive ethyl campholenate, and utilising MM. Bouveault and Blanc's method of reduction. He finds that the  $\beta$ -campholenol isomer of borneol has the constitution—



**New Dinaphthopyranic Phenols.**—R. Fosse.—The author discovers the curious property of pyranic phenols, which apparently belongs to the whole family, that they are insoluble in the aqueous alkalis and soluble in alcoholic alkalis. This anomaly of the phenolic function can be explained by admitting that the basicity of the pyranic oxygen neutralises the acidity of the phenolic hydroxyl to form a kind of salt stable in alkalis in solution in water; but, on the contrary, decomposable by alkalis in presence of alcohol. He also describes the new phenols which result from the copulation of dinaphthopyryl salts with the three ortho-, meta-, and para-cresols.

**Alcoyl-allyl-ketones.**—E. E. Blaise.—The author synthesises the alcoyl-allyl-ketones by condensation of allyl iodide with the nitriles in the presence of zinc. The condensation should be effected at 0° and contact prolonged for about twenty-four hours. The reaction is expressed by the following equations:—



**Oxalcoyl-ethylenic Carbides and Acids.**—Charles Moureu.—The author isolates in a pure state a series of oxalcoyl-ethylenic acids,  $\text{RC}(\text{OR}')=\text{CH}-\text{CO}_2\text{H}$ , and oxalcoyl-ethylenic carbides,  $\text{RC}(\text{OR}')=\text{CH}_2$ , and investigates the various decompositions of these compounds. Further, he condenses certain alcohols with phenyl-acetylene.

**Reduction of Nitrobenzoic Acetals and Acids.**—P. Freundler.—An investigation of the reduction of *o*-nitrobenzylic alcohol and the ether oxides has shown the influence of the substitutions of the radicle on the formation of azoics. The author completes the researches by submitting to the same treatment (powdered zinc, alcohol, and soda) the three acetals and *o*- and *p*-nitrobenzoic acids.

## MISCELLANEOUS.

**Forthcoming Book.**—Messrs. Crosby Lockwood and Son announce for early publication a treatise on "The Oil Fields of Russia and the Russian Petroleum Industry," by A. B. Thompson, A.M.I.M.E. Mr. Thompson, as Chief Engineer and Manager for some years in Russia of one of the leading English companies, has had very wide and exceptional experience in the exploration, exploitation, and management of oil-bearing properties. Besides dealing with these subjects, he has added to the work Notes on the Origin of Petroleum in Russia, and a translation of the Imperial Rules and Regulations concerning Oil Properties. The work is very fully illustrated with diagrams, photographic plates, and maps.

**Presence of Formic Aldehyde in Atmospheric Air.**—H. Henriet.—The author discovers traces of formic aldehyde in the air. This body is a powerful antiseptic, and therefore plays an important part in the purification of the air. It is an important factor in public hygiene, in addition to its action in vegetable physiology. He estimates the formaldehyde in the air by passing the latter, after filtration, over red mercury oxide heated to 250° and comparing the carbon dioxide thus obtained with that normally existing in the air. He finds the quantity varies between 1/100,000 and 5/100,000 of the weight of the air, and is proportional to the exterior temperature.—*Comptes Rendus*, cxxxviii., No. 4.

**Organo-mercuric Compounds of Salicylic Acid.**—G. Buroni.—The basic salicylate of mercury, to which up till now the formula  $C_6H_4 \cdot \overset{O}{\underset{O}{\text{C}}} \cdot Hg$  has been given, should, on the contrary, be looked upon as a compound containing a nuclear atom of mercury; that is  $C_6H_3 \cdot \overset{OH}{\underset{O}{\text{C}}} \cdot \overset{O}{\underset{O}{\text{C}}} \cdot Hg$ , in every respect analogous to Peschi's mercurio-benzoic anhydride. *Oxymercuriosalicylic anhydride*.—This body can be obtained by the known methods, or by boiling a molecule of salicylic acid with 1 molecule of acetate of mercury; the precipitate is dissolved in dilute soda and re-precipitated with  $CO_2$ . It is a white powder, decomposing when heated. The ammonium salt occurs in easily decomposable needles, soluble in water. *Chloro-mercuriosalicylic acid*,  $C_6H_3 \cdot \overset{OH}{\underset{O}{\text{C}}} \cdot \overset{O}{\underset{O}{\text{C}}} \cdot HgCl$ , occurs in needles, soluble in boiling alcohol. It is obtained by the action of acetic acid on the salts. *Bromomercuriosalicylic acid* is prepared in the same manner as the preceding ones.—*Gazz. Chim. Ital.*, vol. xxxii., p. 305.

**Problem of the Systematic Classification of Inorganic Compounds.**—M. Locke.—We know that Abegg and Bödlander look upon the properties of the organic salts as functions of the electro-affinity of the ions in solution (*Zeit. Anorg. Chem.*, vol. xx., p. 453). In a previous paper the author has arrived at results contrary to the views expressed by these chemists (*Am. Chem. Journ.*, vol. xxvii., p. 105). In the case of monovalent metals, a compound is the more soluble as it has less tendency to form complex salts. The more easily it forms ammoniacal compounds, the greater tendency it has to form double salts, and the less easily it is soluble. However, the thalious salts form an exception, for the halogenides, though slightly soluble like the salts of silver, do not form complex ions, thus behaving like the salts of sodium. If we consider the elements of different valencies, the relations which exist between the solubility and facility to form double salts and ammoniacal derivatives are not necessarily in the same sense. Thus with zinc the formation of complex ions seems to be connected with the great solubility of the salts. The author then proceeds to deal with the relations existing between the solubilities of compounds and

the place of the elements in the periodic system. In the chloride, bromide, iodide system, for example, the solubility increases from the chloride to the iodide if the chloride is easily soluble (Na, Ba, K, Fe). If, on the other hand, the chloride is not easily soluble, the solubility of the iodide is almost nil (Ag, Pb, Tl, Hg). In the latter portion of this paper the author deals with the solubility of the alums and the sulphates of the magnesian series:—

## Solubility of the Alums in Grm.-molecules per Litre.

|                       | Al.   | Cr.   | Va.   | Fe.   |
|-----------------------|-------|-------|-------|-------|
| Cs .. ..              | 0.013 | 0.015 | 0.020 | 0.045 |
| Rb .. ..              | 0.059 | 0.078 | 0.177 | 0.293 |
| Tl .. ..              | 0.177 | 0.212 | 0.573 | 0.799 |
| NH <sub>4</sub> .. .. | 0.387 | 0.407 | 1.210 | 1.659 |

With the alums the influence of an element on the solubility of the isomorphous mixtures may be represented by a constant which remains the same for the whole series of alums.—*Zeit. Anorg. Chem.*, vol. xxxiii., p. 58.

## MEETINGS FOR THE WEEK.

- MONDAY, 7th.—Society of Arts, 8. (Cantor Lectures). "Recent Advances in Electro-chemistry," by Bertram Blount, F.I.C.  
 Royal Institution, 5. General Monthly Meeting.  
 Society of Chemical Industry, 8. Presentation to Mr. A. R. Ling. "Observations on Cotton and Nitrated Cotton," by H. de Moenshal. "The Products, and Relative Temperature of Combustion of some Smokeless Powders," by W. Macnab and A. K. Leighton.  
 TUESDAY, 8th.—Royal Institution 5. "Japanese Life and Character," by Ernest Forwell, M.A.  
 WEDNESDAY, 9th.—Society of Arts, 8. "Mechanical Piano Players," by J. W. Coward.  
 THURSDAY, 10th.—Royal Institution, 5. "Electrical Methods of Measuring Temperature," by Prof. H. L. Callendar, F.R.S.  
 Society of Arts, 4.30. "China Grass—its Past, Present, and Future," by Frank Birdwood, B.A.  
 FRIDAY, 11th.—Royal Institution, 9. "The Motion of Viscous Substances," by Prof. Frederick T. Trouton, F.R.S.  
 Physical, 8. (At Royal College of Science, South Kensington). "The Whirling and Transverse Vibrations of Shafts," by Dr. C. Chree, F.R.S. "Notes on Non-homocentric Pencils, and the Shadows produced by them—Part II., Shadows produced by Axially Symmetrical Pencils possessing Spherical Aberration," by W. Bennett. Exhibition of Apparatus by Messrs. Pye and Co., Cambridge.  
 SATURDAY, 12th.—Royal Institution, 3. "The Life and Work of Stokes," by Right Hon. Lord Rayleigh, F.R.S.

The following Reminders of Stock are to be sold far below value:—

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CONTENTS.

| ARTICLES:—                                                                                                  | PAGE |
|-------------------------------------------------------------------------------------------------------------|------|
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S.                                 | 121  |
| On the Law of Thermic Constants, and the Heat of Formation of Compounds of Barium, by D. Tommasi            | 123  |
| The Chemical Analysis of two Rare Minerals from the Caucasus, in the Batoum District, by G. P. Tchernik     | 123  |
| Constant-standards Silver Trial-plates, by Edward Matthey, C.B.                                             | 124  |
| Some Applications of the Theory of Electrolysis to the Separation of Metals from one another, by A. Høiland | 125  |
| PROCEEDINGS OF SOCIETIES—                                                                                   |      |
| PHYSICAL SOCIETY.....                                                                                       | 127  |
| INSTITUTE OF CHEMISTRY.....                                                                                 | 128  |
| NOTICES OF BOOKS.....                                                                                       | 129  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES.....                                                                  | 130  |
| MISCELLANEOUS.....                                                                                          | 131  |
| MEETINGS FOR THE WEEK.....                                                                                  | 132  |

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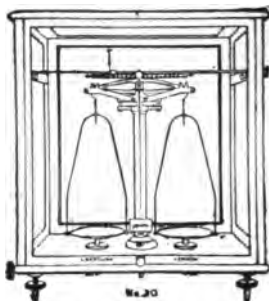
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2311.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.\*

By CHARLES BASKERVILLE, Ph.D., F.C.S.,  
Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 110).

THE imposition upon your good nature practised in the foregoing craves its pardon in an effort to seek a definition for the term *element*. Shall we say, as does Remsen, "an element is a substance made up of atoms of the same kind?" Can we say that it is not? Venable truly says ("Definition of the Element," *loc. cit.*): "An element is best defined by means of its properties." These conceits are not exclusive. The properties are the result of the action of physical forces and chemical affinity, whatever that may be. Certain of the novel atmospheric gases have so far responded but poorly to the latter, as predicted before their discovery by Flawitzsky, Julius Thomsen, and de Boisbaudran in 1887. This necessitates, according to Piccini, our dividing them at once into two classes (*Zeit. Anorg. Chem.*, 1899, xix., 295).

Pattison Muir gives a satisfactory definition ("The Alchemical Essence and the Chemical Element," London, 1894:—"The notion of the elements that has been attained after long continued labour is that of certain distinct kinds of matter, each of which has properties that distinguish it from every other kind of matter, no one of which has been separated into portions unlike the original substance, and which combine together to produce new kinds of matter that are called compounds." The following simpler definition has finally served as my guide:—*An element is that which has not been decomposed, so far as we are aware, into any thing other than itself.* In short, it is consistent.

It is well to stop occasionally and take stock. The Daltonian centenary could not but be an opportune time. Stable certified securities are not enumerated in the list which follows. Having in mind the second chapter of the first book of Chronicles, certain so-called elements are mentioned; for yttrium begat cerium, and cerium begat lanthanum, and lanthanum begat samarium and didymium, and didymium begat neodidymium and praseodidymium, and praseodidymium begat  $\alpha$ - and  $\beta$ -praseodidymium, "and so weiter."

Unpractised as a reading clerk, I shall spare you the strain of hearing this long list of elements on probation, but submit for leisure perusal printed copies.

From the table have been omitted urstoff, protyle (Crookes), electrons (Lodge), corpuscles (J. J. Thomsen), and pantogen (Hinrichs). It appeared also unnecessary to incorporate phlogiston, nitricum (the imaginary body, thought by Berzelius united with oxygen to form nitrogen), and aræon (ponderable caloric). According to Meissner, hydrochloric acid is composed of two equivalents of oxygen, one of water, combined with aræon, and the imaginary radical murium (*vide* Bolton). Often alloys have been prepared and given names like the elements, "magnalium" for example. These are omitted also. Otherwise, I have purposely included every suggestion of an element I could obtain. The summary, while doubtless deficient, may secure an historical vindication.

What shall we do with these numerous aspirants whose recognition is urged? "These elements perplex us in our

researches, baffle us in our speculations, and haunt us in our very dreams. They stretch like an unknown sea before us, mocking, mystifying, and murmuring strange revelations and possibilities," said Crookes referring to the rare earths. Some have been verified, many unverified; some are true, some are false. Without doubt some have been presented without sufficient stage setting, yet the good faith of many can not be questioned. In fact, from this list, as one reads, he perceives the whole gamut of scientific emotions. There he may find the tragedies of elemental pretension, the comedies, yea, the very farces.

We need not look far to ascertain explanations for certain incorrect conclusions. The extreme rarity of the minerals in which many of the tentative elements have been detected, the excessively small percentages of the new ingredients, and the extraordinary difficulties attending their separation from known and unknown substances, combine to render the investigations laborious, protracted, and costly. De Boisbaudran required 2400 kilograms of zinc blende for 62 grms. of gallium. Ramsay (*Zeit. Phys. Chem.*, 1903, xlv., 74) has shown one part of krypton in twenty million volumes of air, while a like amount of xenon requires one hundred and seventy million. How patiently and persistently that modest Parisian couple followed Becquerel's rays!

Furthermore, when one feels that he has obtained something novel, the absolute proof is fraught with difficulties and uncertainties. We have decided to define an element by its properties. The alterations produced in the properties of the most characteristic elements by the presence of small amounts of foreign substances are evident in steel. The influence of arsenic upon the conductivity of copper is well known, and Le Bon (*Comptes Rendus*, 1900, cxxxi., 706) has recently shown that traces of magnesium (one part in 14,000) in mercury cause the latter to decompose water and to oxidise rapidly in the air at ordinary temperatures. Thorium with less than a trace of actinium produces an auto-photograph.

This point can not be too strongly stressed in the rare earth field. One who has wrought with thorium dioxide well knows the influence a small amount of cerium has upon its solubility. The conflicting statements in the literature as to the colours of the oxides of the complexes, neodidymium and praseodidymium, cause one to wonder if different researchers have had the same hæcceity.

An appeal to the spectroscope is of course in the minds of all my hearers.

It was once supposed that each element has its characteristic spectrum which remained the same under all circumstances. Keeler calls attention to modern investigations which have shown that the same element can have entirely different spectra (*Scientific American Supplement*, 1894, lxxxviii., 977, and *Popular Astronomy*). For example, oxygen may be caused to have five different spectra, nitrogen two, &c. In fact, there is no indication in the appearance of the spectra that they belong to the same substance; yet through the result of the work of Rydberg, Kayser, Runge and Precht, series of groups of lines are had which satisfy mathematical formulæ.

"It was proposed by de Gramont, at the International Congress in Paris, in 1900, and agreed, that no new substance should be described as an element until its spark spectrum had been measured and shown to be different from that of every other known form of matter."

As Hartley remarks (Address before the Chemical Section of the British Association, Southport, 1903):—"This appears to me to have been one of the most important transactions of the Congress." Radium was the first to be tested by this rule (Runge and Precht, *Ann. Physik.*, 1903, [4], xii., 407). Exner and Haschek obtained 1193 spark and 257 arc lines for Demarçay's europium. It must not be forgotten, however, that, by overlapping, lines in mixtures may be masked or appear, which are absent in those bodies of the highest state of purity. It must not be forgotten that pressure influences the spectrum, usually producing a broadening of the lines, as shown by Schuster ("British Association Report," 1880, 275; *vide* also

\* Address of the Vice-President of Section C (Chemistry) of the American Association for the Advancement of Science, St. Louis Meeting, December 28, 1903.

Lockyer and Frankland, *Proc. Roy. Soc.*, 1869, xxvii., 288), and that it may occur symmetrically or only towards the least refrangible red. Lest we forget, the spectroscope failed a long time to show radium, and we knew it was there. It must not be forgotten, as G. Krüss has shown, that the "influence of temperature cannot be neglected and ignored, but must be considered by every chemist who wishes to make correct spectroscopic observations" ("The Influence of Temperature upon the Spectrum—Analytical Observations and Measurements," *Liebig's Annalen*, ccxxxviii., 57; *CHEMICAL NEWS*, lvi., 51). It is well known to spectroscopists that band spectra are obtained at temperatures intermediate between those required for the production of continuous spectra and line spectra ("Spectrum Analysis," Landauer, English translation by Tingle, p. 70). The explanations of these facts do not concern us at present.

It has been shown by the researches of Newton, Dale, Gladstone, Jamin, Schrauff, Landolt, and others that the refractive power increases in all liquids, except in water, between 0° and 4° with the increase of density—that is, with decrease of temperature. Rydberg showed that various solid bodies, such as quartz and aragonite, follow the same law. There are some exceptions, however. Among these is glass, as proved by Arago and Neumann prior to Rydberg. "On a rise of temperature, all phenomena of absorption or emission are displaced toward the violet with the glass prisms, but toward the red with quartz prisms. These displacements are the greater the more refrangible the region of the spectrum in which they occur." As a result of a large numbers of observations, Krüss learned that by a variation of 25°, marked changes would be observed in the spectroscopic lines. From a table given, it could be seen that errors may spring from neglect of the temperature (of the instrument?) in stating wave-lengths, since a rise of 5° is sufficient to transfer the D<sub>1</sub> to the position D<sub>2</sub>. Roscoe obtained an entirely new spectrum with the metal sodium, whereby it appears that this metal exists in a gaseous state in four different degrees of aggregation, as a simple molecule, and as three or four or eight molecules together.

Grünwald ("Über das Wasserspectrum, das Hydrogen und Oxygenspectrum," *Phil. Mag.*, 1887, xxiv., 304; "Math. Spectralanalyse des Magnesiums und der Kohle," *Monatshefte für Chemie*, viii., 650; "Math. Spectralanalyse des Kadmiums," *Monatshefte für Chemie*, ix., 956) in a series of papers on his theory of spectrum analysis endeavours "to discover relations between the spectra and thus to arrive at simpler, if not fundamental, 'elements.'" He came to the conclusion that "all the so-called elements are compounds of the primary elements *a* and *b*" (coronium and helium). Ames (*Am. Chem. Journ.*, 1889, xi., 138), having called attention to the use of uncorrected data by Grünwald, remarks:—"The concave grating gives the only accurate method of determining the ultra-violet wave-lengths of the elements, and as a consequence of not using it, most of the tables of wave-lengths so far published are not of much value."

Hutchins and Holden, after a comparative study of the arc spectra of metals and the sun with a twenty-one-foot focal Rowland grating, state ("On the Existence of Certain Elements, Together with the Discovery of Platinum in the Sun," *Am. Journ. Sci.*; *Sci. Am. Supp.*, 1888, xxv., 628):—"We are convinced that there is much in the whole matter of coincidences of metallic and solar lines that needs re-examination; that something more than the mere coincidence of two or three lines out of many is necessary to establish even the probability of the presence of a metal in the sun. With the best instruments the violet portion of the solar spectrum is found to be so thickly set with fine lines that, if a metallic line were projected upon it at random, in many places the chances for a coincidence would be even, and coincidences could not fail to occur in case of such metals as cerium and vanadium, which give hundreds of lines in the arc.

"Moreover, a high dispersion shows that very few lines

of metals are simple and short, but, on the contrary, winged and nebulous, and complicated by a great variety of reversal phenomena. A 'line' is sometimes half an inch wide on the photographic plate, or it may be split into ten by reversals."

Lockyer maintained that the lines of certain substances vary not only in length and in number, but also in brilliancy and in breadth, depending upon the quantity of the substance as well as temperature (*Roy. Soc. Proc.*, lxi., 148, 183; *CHEMICAL NEWS*, lxxix., 145). Being unable to decompose the elements in the laboratory, he studied the spectra of the stars. The spectra of the colder stars (*CHEMICAL NEWS*, lxxix., 147) show many more metals but no metalloids, whereas the coldest stars, *α Orionis*, show the Crookes spectrum of metalloids which are compounds. None of the metalloids are found in the spectrum of the sun. Over 100,000 visual observations and 2000 photographs were made in the researches.

Livinge (Address before the Chemical Section of the British Association; *Scientific American Supplement*, 1882, xiv., 356), as the result of the work of Young, Dewar, Fievez, and himself on the spectrum of the sun, by which some lines were resolved with a new instrument, which they before had not been able to devise, comments on Lockyer's work. That the coincidence of rays emitted by different chemical elements, especially when developed in the spark of a powerful induction coil, and the high temperature of the sun and stars, gives evidence of a common element in the composition of the metals which produce the coincident rays. "This result cannot fail to shake our belief, if we had any, in the existence of any common constituent in the chemical elements, but it does not touch the evidence which the spectroscope affords us that many of our elements, in the state in which we know them, may have a very complex molecular structure."

Hartley (*loc. cit.*) in his recent admirable address said:—"I have always experienced great difficulty in accepting the view that because the spectrum of an element contained a line or lines in it which were coincident with a line or lines in another element, it was evidence of the dissociation of the elements into simpler forms of matter. In my opinion evidence of the compound nature of the elements has never been obtained from the coincidence of a line or lines exclusively belonging to the spectrum of one element with a line or lines in the spectrum exclusively belonging to another element. This view is based upon the following grounds:—(1) Because the coincidences have generally been shown to be only apparent, and have never been proved to be real; (2) because the great difficulty of obtaining one kind of matter entirely free from every other kind of matter is so great that where coincident lines occur in the spectra of what have been believed to be elementary substances, they have been shown from time to time to be caused by traces of foreign matter, such as by chemists are commonly termed impurities; (3) no instance has ever been recorded of any homologous group of lines belonging to one element occurring in the spectrum of another, except and alone where the one has been shown to constitute an impurity in the other; as, for instance, where the triplet of zinc is found in cadmium and the triplet of cadmium in zinc; the three strongest lines in the quintuple group of magnesium is graphite, and so on. The latest elucidation of the cause of coincidences of this kind arises out of a tabulated record from the wave-length measurements of about three thousand lines in the spectra of sixteen elements made by Adeney and myself. The instances where lines appeared to coincide were extremely rare; but there was one remarkable case of a group of lines in the spectrum of copper which appeared to be common to tellurium; also lines in indium, tin, antimony, and bismuth, which seemed to have an origin in common with those of tellurium.

The last sentence presents the point I wish to emphasise. Tellurium has long obtruded itself before a satisfactory vision of the natural system. The table appended alone recites not a few efforts to obtain the contaminating

constituent of tellurium which *a priori* is present from Hartley's observations (see also Grünwald, 1889, table). The fractionation of a rubidium-cæsium mixture, perhaps, is a simpler problem than that confronting Pellini (*Gazz. Chim. Ital.*, xxxiii., 11, 35), who reports a definite amount of an element with a high atomic weight (about 214) similar to, and associated with, tellurium.

What has been said applies especially to the elements of the rare earth class—"asteroids of the terrestrial family"—as phrased by Crookes. Many of them have not been secured with sufficient purity to claim an inherent spectrum; further, the spectra attributed have not been obtained under uniform conditions.

(To be continued).

# ON THE LAW OF THERMIC CONSTANTS, AND THE HEAT OF FORMATION OF COMPOUNDS OF BARIUM.

By D. TOMMASI.

M. GUNTZ has recently determined the heat of oxidation of barium (*Comptes Rendus*, May 4, 1903), and found  $\text{Ba} + \text{O} = \text{BaO}$  solid, 133.4 cal. This figure enables us easily to calculate the actual heat of formation of dissolved chloride of barium, which is equal to 198.8 cal.

To obtain the thermic constant of barium it suffices to subtract from the heat of formation of dissolved chloride of potassium the heat of formation of the chloride of barium, also dissolved:  $2 \times 100.8 - 198.8 = 2.8$  cal. Knowing the thermic constant of barium (2.8 cal.), it is easy to determine, with the help of the law of thermic constants, the heat of formation of all the soluble compounds of this metal. (For further details see *Bull. Soc. Chim.*, 1883, pp. 384, 390, and 602; and the "Traité d'Electrochimie," by D. Tommasi, p. 856).

## Heats of Formation of the Compounds of Barium.

|                            | Calculated.<br>Calories. | Found.<br>Calories. |
|----------------------------|--------------------------|---------------------|
| Bromide of barium .. .. .  | 179.2                    | 179.2               |
| Iodide " .. .. .           | 146.6                    | 146.6               |
| Nitrate " .. .. .          | 189.4                    | 189.2               |
| Perchlorate " .. .. .      | 190.0                    | 190.4               |
| Chlorate " .. .. .         | 189.2                    | 189.0               |
| Hydrate " .. .. .          | 161.8                    | 161.4               |
| Formate " .. .. .          | 188.6                    | 188.4               |
| Acetate " .. .. .          | 188.4                    | 188.2               |
| Ethylsulphate of barium .. | 189.2                    | 189.2               |
| Glycolate " .. .. .        | 189.2                    | 189.2               |
| Benzinosulphate " .. .. .  | 188.6                    | 188.8               |
| Picrate " .. .. .          | 188.6                    | 189.0               |

The following are the heats of formation of some compounds of barium which have not yet been determined experimentally, but which can be calculated by the law of thermic constants. This law, which I discovered in the year 1882, may be stated as follows:—When a metal is substituted for another in a saline solution, the heat given off by each metal is always the same, no matter what may be the nature of the acid radical present in the salt.

| Compounds.                   | Calories. |
|------------------------------|-----------|
| Bichromate of barium .. .. . | 188.6     |
| Butyrate " .. .. .           | 139.0     |
| Chloracetate " .. .. .       | 190.4     |
| Trichloracetate " .. .. .    | 189.8     |
| Amidobenzoate " .. .. .      | 180.2     |
| Nitrobenzoate " .. .. .      | 187.2     |
| Benzoate " .. .. .           | 188.6     |
| Lactate " .. .. .            | 188.6     |

\* The heat of formation of chloride of potassium, determined by Thomsen.

Thus we see that, by knowing the heat of formation of a single compound of barium, it is possible, thanks to the law of thermic constants, to determine with the greatest accuracy the heats of formation of all the other soluble compounds of barium.

It is very evident that what I have just stated for compounds of barium can be applied, without exception, to all the soluble mineral and organic salts.—*Bull. Soc. Chim.*, Series 3, xxix., No. 16.

## THE CHEMICAL ANALYSIS OF TWO RARE MINERALS FROM THE CAUCASUS, IN THE BATOUM DISTRICT.

By G. P. TCHERNIK.

THE author has explored the delta of the river Tchorokh, and has collected several minerals from the country traversed by this river or its affluents. The complete analysis of two rare minerals is given below. They were found in a rock of granitic nature, formed of felspar of a light colour in fairly large crystals, with large white flakes of mica, and dark coloured quartz—a mixture in which the felspar is predominant.

One of these minerals occurs in plates of 2 to 2.5 m.m. thick, separated one from the other by thin layers of brown oxide of iron. Their structure is crystalline, though their form has not been determined; their lustre is greasy, hardness = 5.5, density = 5.485; their colour is velvet-black; and their fracture conchoidal. When heated, they split up, become opaque, and take a brown colour; after heating strongly, their density diminishes by about 5 per cent; in the blowpipe flame, they fuse on the edges with difficulty, and give a dark greyish coloured glass. The borax bead is dirty red in the oxidising flame, and dark green in the reducing flame; the phosphorus-salt bead is greenish in both flames. The analysis has been made minutely, and is as follows:—

|                                        | Per cent. |
|----------------------------------------|-----------|
| Ta <sub>2</sub> O <sub>5</sub> .. .. . | 26.88     |
| Nb <sub>2</sub> O <sub>5</sub> .. .. . | 33.80     |
| Y <sub>2</sub> O <sub>3</sub> .. .. .  | 6.15      |
| Er <sub>2</sub> O <sub>3</sub> .. .. . | 4.22      |
| Ce <sub>2</sub> O <sub>3</sub> .. .. . | 3.82      |
| La <sub>2</sub> O <sub>3</sub> .. .. . | 1.07      |
| Di <sub>2</sub> O <sub>3</sub> .. .. . | 0.74      |
| ThO <sub>2</sub> .. .. .               | 3.23      |
| ZrO <sub>2</sub> .. .. .               | 2.17      |
| U <sub>2</sub> O <sub>3</sub> .. .. .  | 4.37      |
| FeO .. .. .                            | 7.36      |
| MnO .. .. .                            | traces    |
| CaO .. .. .                            | 0.94      |
| TiO <sub>2</sub> .. .. .               | 0.60      |
| TeO <sub>3</sub> .. .. .               | 1.90      |
| SnO <sub>2</sub> .. .. .               | traces    |
| MgO .. .. .                            | traces    |
| K <sub>2</sub> O .. .. .               | 0.48      |
| Na <sub>2</sub> O .. .. .              | 0.80      |
| Al <sub>2</sub> O <sub>3</sub> .. .. . | 0.25      |
| SiO <sub>2</sub> .. .. .               | 0.22      |
| H <sub>2</sub> O .. .. .               | 99.00     |

A comparison with the analyses of analogous minerals points to a variety of samarskite.

The second of these minerals occurs in short prisms, badly formed and striated in some places; their colour is steel-grey, with a metallic lustre, more or less tarnished in some parts; it is opaque in the mass, but is translucent at the edges; the fracture is irregular, the hardness 6, and density 5.396; their composition is much less complex than that of the preceding one. The results of the analysis are:—

|                                        | Per cent. |
|----------------------------------------|-----------|
| Nb <sub>2</sub> O <sub>5</sub> .. .. . | 62.80     |
| Ta <sub>2</sub> O <sub>5</sub> .. .. . | 19.72     |
| FeO .. .. .                            | 11.16     |
| MnO .. .. .                            | 2.85      |
| TeO <sub>3</sub> .. .. .               | 0.14      |
| SnO <sub>2</sub> .. .. .               | 0.60      |
| ZrO <sub>2</sub> .. .. .               | 0.54      |
| SiO <sub>2</sub> .. .. .               | traces    |
| Al <sub>2</sub> O <sub>3</sub> .. .. . | traces    |
|                                        | 97.81     |

This mineral appears to be a variety of columbite or niobite.—*Journ. Soc. Phys. Chim. R.*, vol. xxxiv., p. 684.

#### CONSTANT-STANDARD SILVER TRIAL-PLATES.\*

By EDWARD MATTHEY,  
C.B., F.S.A., F.C.S., Assoc. Roy. Sch. Mines.

REFERRING to my paper communicated to the Royal Society, February 16, 1894, and read March 15, 1894 (*Proc. Roy. Soc.*, 1894, lv., p. 265), in which it was shown that moderately sized plates of sterling silver of a uniform

Weighing 6.655 kilograms.

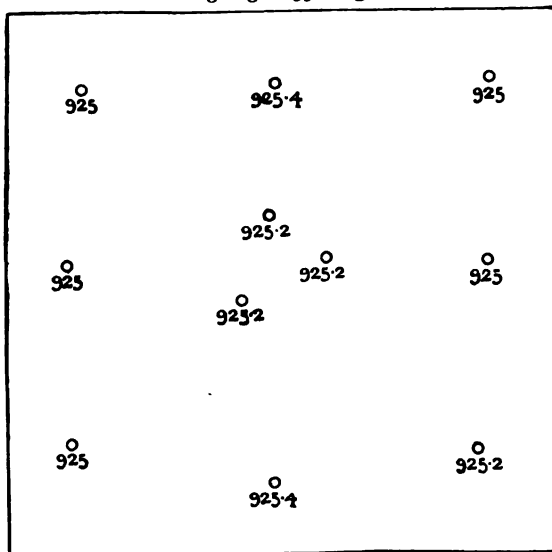


FIG. 1.

Both 75 c.m. by 75 c.m. and 1 m.m. in thickness.

states:—"From the foregoing remarks it will be evident that it is impossible to cast a standard silver plate or bar of uniform composition, and it was necessary therefore to resort to an artifice in order to obtain a standard trial-plate of the required dimensions." ("Fourth Annual Report," Deputy Master of Mint, 1873, pp. 44-46.)

The means adopted by him were to cast 1000 ozs. of standard silver into a skillet-mould 30 c.m. long, 25 c.m. wide, and 5 c.m. broad, to plane off 4 m.m. from its surface, and to roll the planed skillet to 1.8 m.m. thickness, a sheet being produced 1.5 m. long, 45 cm. wide. From this the portion was cut which showed constant results about 925, but which varied from 924.6-925.1; and the rest of the plate, which varied from 924 (lowest) to 928.4 (highest), was abandoned. He shows all these results by diagrams accompanying his memoranda. The portion of available constant standard so cut out from the 1000 oz. sheet weighed 104 ozs., about one-tenth of the whole plate. And this 104 ozs. (= 3.230 kilograms.) formed the mint trial plate from a mass of 1000 ozs. (= 31.103 kilograms.) specially cast for the purpose.

Notwithstanding that many experiments were subsequently made by Prof. Roberts-Austen to find a means of obtaining a constant standard trial-plate, in 1899 he was compelled to resort to what he calls the "cumbrous expedient" of 1873. His statement is:—"None of the

Weighing 6.624 kilograms.

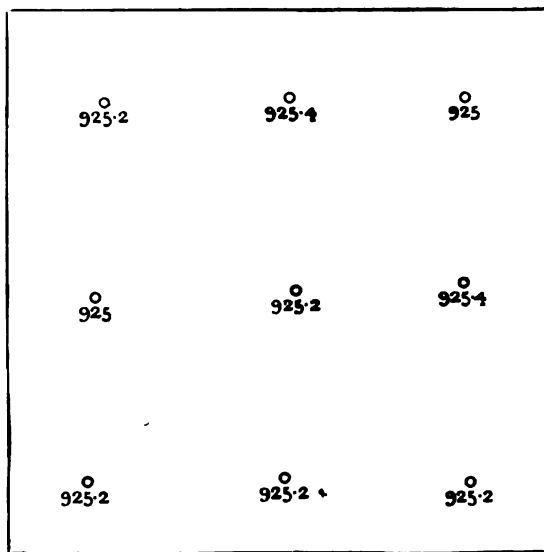


FIG. 2.

standard could be obtained by casting from thin castings, my attention since then was drawn to the difficulty of casting larger quantities than those described in that paper, which were only of an average weight of 4 to 5 kilograms per plate, and to the desirability of obtaining a large plate, say of some 8 or 10 kilograms in weight without difficulty, and I have therefore resumed my attention towards effecting this. It appears that considerable difficulties have been experienced with regard to obtaining large plates of constant standard.

In the Royal Mint Report of 1873 is a memorandum appended by Prof. W. Chandler Roberts, which refers to a series of well-known experiments with regard to obtaining a constant alloy of 0.900 silver by Levol in the Paris Mint, he himself being at the time engaged in the preparation of a standard silver trial-plate. Prof. Chandler Roberts

results were satisfactory, and eventually, after no less than 31 plates had been cast without success, recourse was had to the method adopted in 1873, of casting a large mass of metal and detaching a particular portion, which proved by assay to be of approximately uniform standard." ("Thirtieth Annual Report," Deputy Master of Mint, 1899, pp. 69, 70.)

The casting of the standard silver into thin instead of into the thick moulds ordinarily employed having been attended with such excellent results (*vide* my paper of February 16, 1894, before referred to), I was induced to believe that it must be due to the more rapid cooling of the metal by which liquation was arrested; so that if the standard silver were cast into moulds sufficiently cooled, liquation might be induced to disappear altogether. In order then to overcome the difficulty of obtaining the standard trial-plates in larger sizes, I commenced by casting quantities of not less than 8 kilograms into a mould cooled externally by ice, and also by freezing mixtures as

\* A Paper read before the Royal Society, February 11, 1904.

low as  $10^{\circ}$  C., and by these means I obtained most encouraging results—results which confirmed me in the supposition that by cooling with rapidity there is less time for liquation, which appears to be the direct converse of what has been supposed hitherto, viz.: “that a uniformity of standard was best attained by slow and uniform cooling” (*vide* memorandum already referred to in Mint Report, 1873). But although I thus obtained exceedingly good

Another cast simply into a cold mould, and rolled to a thickness of 1 m.m., gave the results shown in Fig. 5.

This only weighed 3.640 kilograms., is without any wave of variation, and is absolutely constant.

The hitherto accepted theory “that the molecular rearrangement is comparatively slight if the mass of metal is slowly and uniformly solidified” (“Fourth Annual Report,” Deputy Master of Mint, 1873, p. 46) is contradicted by

Weighing 7.870 kilograms.

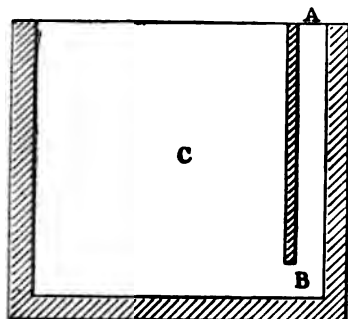


FIG. 3.—Rough Section of Cast-iron Mould employed.

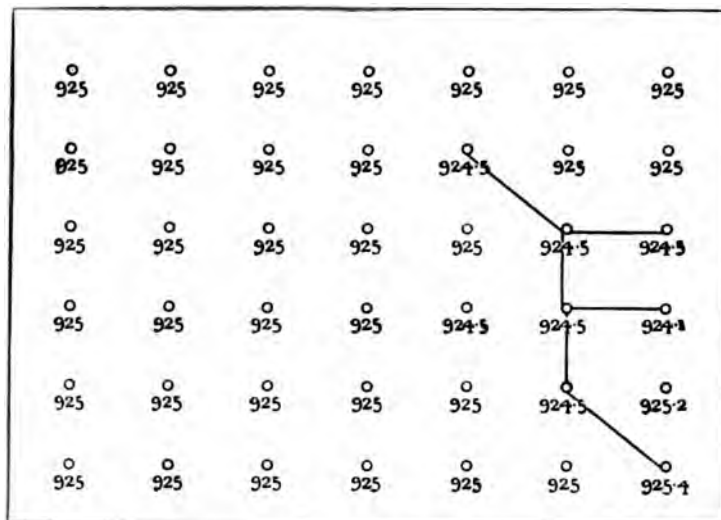


FIG. 4.

75 c.m. by 90 c.m. and 1 m.m. in thickness.

results (Figs. 1 and 2) I was not satisfied that this was the best way of producing a constant-standard plate.

I therefore adopted a different method of casting the standard silver. Instead of pouring the melted alloy into a mould from the top, I poured it into a mould by which the skillet was produced from the bottom (see Fig. 3).

By pouring the alloy into the gate A, the metal passes by B into the space C. And instead of cooling the mould

the results I have obtained; and the results all bear out the fact that there is little or no difficulty in obtaining an 8 or 10-kilogram plate of constant-standard silver, or even more if necessary.

## SOME APPLICATIONS OF THE THEORY OF ELECTROLYSIS TO THE SEPARATION OF METALS FROM ONE ANOTHER.\*

By A. HOLLARD.

(Concluded from p. 113).

### II. Influence of the Nature of the Cathode.

By suitably choosing the metal for the cathode, it is possible to avoid completely the liberation of hydrogen, even in strongly acid solution, and thus to increase sufficiently the conductivity of the bath so that the separation of the metals becomes possible. Moreover, if the metal of the cathode raises the polarising E.M.F. of hydrogen above the polarising E.M.F. of the metal M it is proposed to deposit, at the same time keeping it below the polarising E.M.F.'s of the metals which are to remain in the bath, it becomes impossible, as a rule, for these metals to be deposited. In fact, if one does reach the E.M.F. at which they are deposited, this potential, being higher than that of hydrogen, causes in acid solution a sufficiently abundant evolution of gas to prevent their deposit. If evolution of hydrogen occurs, it shows that the E.M.F. being used is too great.

The above requires amplifying a little.

Let us call to mind first that in electrolytic analysis the metals are divided into two principal classes, according as

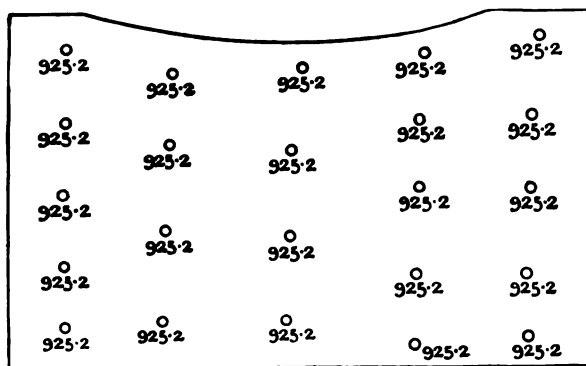


FIG. 5.

by ice or freezing mixture I used the mould simply cold. I have obtained excellent results by this method. Subjoined is one of the plates produced rolled to 1 m.m. thick, measuring 90 c.m.  $\times$  75 c.m. and weighing 8.700 kilograms. Trimming the rough edges from the plate about 1 c.m., the results came out as under, showing a constancy of 925 nearly all over the plate. I have drawn a line where the only wave of lower variation occurs (Fig. 4).

\* A Paper read before the Faraday Society, February 2, 1904.

they are or are not capable of being deposited on the cathode in strongly acid solution.

The metals which cannot deposit themselves in strongly acid solutions are those which, in order to cover the cathode, require potentials higher than that at which hydrogen begins to evolve; one sees then that, under the influence of these high tensions, a strongly acid solution—that is, one containing a large proportion of  $H^+$  ions—gives rise to such an abundant evolution of hydrogen at the cathode, that all precipitation of metal there becomes impossible. On the other hand, metals which can be deposited on the cathode in strongly acid solution are those which require for this precipitation potentials lower than the polarisation potential of hydrogen; their deposition is not then interfered with by hydrogen.

If we call the polarisation potential of hydrogen zero, we shall have the following values for the polarisation potentials of the principal metals (Nernst and Wilmore, *Zeit. f. Elektrochem.*, November 8, 1900):—

TABLE I.

Metals which cannot be Deposited in Strongly Acid Solution.

|      |    |    |    |    |       |
|------|----|----|----|----|-------|
| Zn.. | .. | .. | .. | .. | +0.77 |
| Cd.. | .. | .. | .. | .. | +0.42 |
| Fe.. | .. | .. | .. | .. | +0.34 |
| Co   | }  | .. | .. | .. | +0.23 |
| Ni   |    | .. | .. | .. |       |
| Sn.. | .. | .. | .. | .. | +0.19 |
| Pb.. | .. | .. | .. | .. | +0.15 |
| H..  | .. | .. | .. | .. | 0.00  |

Metals which can be Deposited in Strongly Acid Solutions.

|      |    |    |    |    |       |
|------|----|----|----|----|-------|
| As.. | .. | .. | .. | .. | 0.29  |
| Cu.. | .. | .. | .. | .. | -0.33 |
| Bi.. | .. | .. | .. | .. | -0.39 |
| Sb.. | .. | .. | .. | .. | -0.47 |
| Hg.. | .. | .. | .. | .. | -0.75 |
| Ag.. | .. | .. | .. | .. | -0.77 |
| Pd.. | .. | .. | .. | .. | -0.79 |
| Pt.. | .. | .. | .. | .. | -0.86 |
| Au.. | .. | .. | .. | .. | -1.08 |

As one sees, the division of the metals into two groups has for its basis the position occupied by hydrogen in the table of the polarisation potentials of the metals. But this position is only fixed in the practice of electrolytic analysis because platinum cathodes are always used. In fact, hydrogen has—as shown by Caspari (*Zeit. f. r. Phys. Chem.*, 1899, xxx., 1889)—a polarisation potential varying with the metal of the cathode. If one then uses, as we have done, cathodes of other metals besides platinum, the polarisation potential of hydrogen takes another position in the above table, causing a certain number of metals to pass from one group into the other.

By appropriately choosing the cathode metal, it is evidently possible to achieve the separation of two metals of the same group whose polarisation potentials are too near to be separated with a platinum cathode; a metal is chosen giving to hydrogen a polarisation potential intermediate between those of the two elements to be separated. In a strongly acid solution the element with the lowest polarisation potential will be precipitated, the bath being made a good conductor by the non-evolution of hydrogen at the cathode.

In order to choose the most suitable metal, the following table, due to Caspari, must be consulted; this gives the values of the polarisation potential of hydrogen when disengaged on different metals taken as cathode in normal sulphuric acid. (See Table II.).

But it is not enough to choose as cathode a metal which has the property of raising the polarisation potential of hydrogen above the polarisation potential of the metal to be precipitated, for this property of the cathode metal disappears when it is covered by the precipitated element, and

TABLE II.

|                            |    |    |    |    |      |
|----------------------------|----|----|----|----|------|
| Pt, platinised             | .. | .. | .. | .. | 0.00 |
| Au                         | .. | .. | .. | .. | 0.02 |
| Fe (in NaOH)               | .. | .. | .. | .. | 0.08 |
| Pt, polished               | .. | .. | .. | .. | 0.09 |
| Ag                         | .. | .. | .. | .. | 0.15 |
| Ni                         | .. | .. | .. | .. | 0.21 |
| Cu                         | .. | .. | .. | .. | 0.23 |
| Pd                         | .. | .. | .. | .. | 0.46 |
| Cd                         | .. | .. | .. | .. | 0.48 |
| Sn                         | .. | .. | .. | .. | 0.53 |
| Pb                         | .. | .. | .. | .. | 0.64 |
| Zn (in acid zinc solution) | .. | .. | .. | .. | 0.70 |
| Hg                         | .. | .. | .. | .. | 0.78 |
| Cu, amalgamated            | .. | .. | .. | .. | 0.51 |
| Pb, amalgamated            | .. | .. | .. | .. | 0.54 |
| Cd                         | .. | .. | .. | .. | 0.68 |

then acts as if made entirely of this precipitated element. In order that the deposition may continue, it is necessary that the precipitated metal itself should also possess the property of raising the polarisation potential of hydrogen above its proper value.

By taking cathodes made of the same metal as that to be deposited, it is seen that, according to the preceding table, it is possible to precipitate in strongly acid solutions, not only the metals whose potentials are lower than that of hydrogen (Table I.), but lead, tin, and cadmium. In fact, the polarisation potential of lead (+0.15) is notably lower than that of hydrogen when disengaged on a lead cathode, and the same can be said of tin and cadmium.

As an application of this new method of analytical separation, we will give the separation of zinc and cadmium—metals which we have not been able to separate before with a platinum cathode,\* on account of the small difference of their polarisation potentials. On the other hand, we have been able to carry out the separation with a tin or cadmium cathode in a strongly acid solution. Our cathodes were none other than our platinum-gauze electrodes covered electrolytically with tin or cadmium.

The cadmium or the zinc, in the state of sulphates, were treated with 10 grms. of ammonium sulphate and an excess of 5 c.c. of concentrated sulphuric acid. The solution was diluted to 300 c.c., and a current of 0.3 ampère allowed to pass.

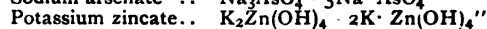
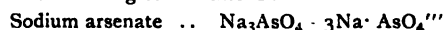
#### Experimental Results.

Quantities weighed.†

| Cd.    | Zn. | Cadmium deposited. |                                                |
|--------|-----|--------------------|------------------------------------------------|
| 0.1991 | 2   | 0.1972             | Cathode of tinned platinum-gauze.              |
| 0.2996 | 2   | 0.3010             |                                                |
| 0.1996 | 1   | 0.2002             |                                                |
| 0.1991 | 2   | 0.1984             | Cathode of platinum-gauze plated with cadmium. |
| 0.2488 | 2   | 0.2484             |                                                |
| 0.1991 | 0   | 0.1968             |                                                |
| 0.2000 | 2   | 0.0002             | Cathode of platinum-gauze.                     |

#### III. Formation of Complex Salts.

One way of preventing a certain number of metals from precipitating themselves on the cathode, is to make them pass into the state of complex salts; that is, salts which dissociate to give, not the metallic ions, but complex ions containing the metal. For example, if we wish to make arsenic or zinc pass into the state of complex salts, we can form the following combinations:—



The complex salts have been studied from the point of

\* Besides using weak acetic acid or cyanide solutions and the feeblest possible currents.

† The cadmium used in the first three experiments contained:—Pb=0.231, Fe=0.000, Zn=0.000 per cent. That used in the three following contained:—Pb=0.245, Fe=0.193, Zn=0.000 per cent. The figures given in the table as "Quantities weighed" differ from the amounts actually weighed by the quantity of impurity present.



view of their application in electrolytic analysis, especially by Frendenberg (*Zeit. Phys. Chem.*, xiii., 97).

As a matter of fact, a certain number of complex anions are partially dissociated into metal ions; the metal can then be deposited on the cathode, but will require an E.M.F. higher than the polarisation potential of the simple salts.

If in a solution of several metals some of them pass into the state of complex salts, then those metals taking the part of non-dissociable complex anions will not separate under the influence of the current, and the metals taking the part of dissociable complex ions will only separate at a higher potential than the polarisation potential of their simple salts.

This is then a means of widening the intervals between the polarisation potentials and of applying the principle of the successive separation of metals by gradual increase of the E.M.F.

The separation of antimony and tin in a concentrated solution of sodium hydrogen sulphide, as indicated by Classen, is an example of the use of complex salts. The tin here passes into the state of a complex anion.

If copper is present with the tin, the copper being slightly soluble in the sodium-hydrogen sulphide, deposits itself with the antimony. This has been shown by us (*Comptes Rendus*, 1896, cxxxiii., 1064; *Ann. de Chim. Analyt.*, 1897, 242).

We avoid this precipitation by adding potassium cyanide to the bath; this causes the copper to pass into the state of a slightly dissociated anion, which is not electrolysed by the potential employed. (For further details see HOLLARD, "Separation and Determination of Antimony by an Electrolytic Method," *Bull. Soc. Chim.*, 1903, xxix., 262).

## PROCEEDINGS OF SOCIETIES.

### PHYSICAL SOCIETY.

Ordinary Meeting, February 26th, 1904.

Dr. R. T. GLAZEBROOK, F.R.S., President, in the Chair.

A NEW Dilatometer, exhibited by Mr. B. BONNIKEN, was described by Mr. B. F. E. KEELING.

The instrument was originally designed for measuring the expansion of balance-wheels of watches, and has latterly been applied to the determination of the coefficient of dilatation of specimens of materials, used in the form of wires about  $1\frac{1}{2}$  inches in length. The increase in length with change in temperature is magnified about 1500 times by means of a chain of accurately mounted gear-wheels, the last one of which moves a pointer over a circularly graduated scale. With an ordinary specimen of steel one degree rise in temperature causes a movement of the pointer over about one-third of a scale-division, and a mean coefficient of expansion of such a substance over a range of  $100^{\circ}$  C. can be obtained to about 1 per cent in a single experiment of five minutes' duration. The instrument has been standardised by using specimens cut from bars which have been examined by Mr. Keeling in the water-bath comparator at the National Physical Laboratory.

The CHAIRMAN expressed his interest in the dilatometer, and said he had had an opportunity of seeing it in use at the National Physical Laboratory. Results were obtained easily and rapidly, and one advantage of the instrument was that it worked satisfactorily with small specimens.

Dr. W. WATSON read a paper on "A Quartz-thread Vertical Force Magnetograph."

The late Dr. Eschenhagen was the first to show that when the moment of inertia of the suspended system in a horizontal-force magnetograph is made very much smaller

than that employed in the ordinary form, it is possible to detect variations in the earth's field of quick period. The design of a satisfactory form of instrument to record rapid variations of the vertical component presents very considerable difficulties. Thus if an attempt is made to reduce to any great extent the moment of inertia, and hence the mass of the balanced magnet in the ordinary form of Lloyd's balance, it is found that irregularities are immediately introduced owing to imperfections or dirt interfering with the free movements of the knife-edge. Further, slight tremors cause the knife-edge to slip about on the plane, and thus the azimuth of the magnet varies. The instrument which the author has designed and constructed resembles, in principle, the quartz-thread gravity balance of Prof. Threlfall. In addition to the advantages derived from the suppression of the knife-edge, the instrument can be simply and accurately compensated for the effects of changes of temperature. The principle of the instrument is to have a magnet suspended on a horizontal quartz fibre kept stretched by means of a spring. The centre of gravity of the magnet and the torsion of the fibre are so adjusted that the axis of the magnet is horizontal. Any variation of the vertical force produces a rotation of the magnet about the fibre which can be suitably recorded by means of a mirror attached to the magnet. The temperature compensation is effected by weighing the magnet on the same side of the axis of the fibre as the south pole. So that the magnetic couple and the couple due to the torsion of the fibre act in the same direction. Hence, since an increase in temperature causes one of these couples to decrease and the other to increase, by suitably adjusting the weight, and therefore the magnitude of the torsion couple, complete compensation can be obtained. The suspended system in the instrument shown at the meeting consists of two magnets 8 c.m. long and 1 m.m. diameter attached by means of small platinum straps to two fused rods of silica which form part of the plate of fused silica forming the mirror. The upper surface of the mirror is platinised. The fixed mirror is supported on the base of the instrument, and is capable of adjustment. The instrument has been tested at the National Physical Laboratory, and it is proposed to set up one of the instruments in the new Magnetic Observatory.

Mr. C. V. BOYS congratulated the author, and said he had had the opportunity and privilege of watching the growth of the instrument. It showed that the properties of quartz-fibres were even more perfect than anything he himself had ever claimed for them. The fact that it was possible to design and construct such an instrument in a physical laboratory showed an advance in scientific education. The theory of the instrument had been carefully worked out, and everything that had to be done practically had been done in the best possible way.

Prof. J. D. EVERETT expressed his interest in the paper, and said there was no novelty in using a horizontal fibre for a control. Horizontal wires were so used in Kelvin's portable electrometer and in the gauge of an ordinary quadrant electrometer. He thought the instrument would be improved by increasing the number of needles.

Dr. J. A. HARKER asked the author for the ordinary working sensitiveness of the instrument. When working with magnetographs of the Eschenhagen type, he had found they became unstable when the sensitiveness was increased to about three times the Kew standard sensitiveness.

Mr. T. H. BLAKESLEY asked for the period of vibration of the suspended system.

Mr. J. R. ASHWORTH sent a letter which was read by the CHAIRMAN, stating that the temperature compensation might be effected by using drawn steel wire magnets with an incremental temperature coefficient. Magnets can now be made to have, within limits, any assigned temperature coefficient (from negative, through zero, to positive).

The CHAIRMAN congratulated the author upon his success, and said that he was glad that Dr. Watson had

been able to obtain assistance from the National Physical Laboratory.

Dr. WATSON said he did not think that the magnets suggested by Mr. Ashworth would be of much advantage for vertical-force magnetographs, as there would have to be a gravity-bob in all cases in order to adjust the centre of gravity of the moving system. They might, however, be of use in a horizontal-force instrument. Replying to Dr. Harker, he said that 1 c.m. on the records corresponded to 87, whereas 1 c.m. on the Kew records represented 507. Replying to Mr. Blakesley, he said the period of vibration was large, but it was not necessarily important to have as long a period as possible. With regard to Prof. Everett's remarks, he pointed out that all metallic wires were ruled out of the question on account of elastic fatigue. He had used two magnets only because of the experimental difficulties in attaching them to their support.

A paper by Mr. G. W. WALKER, "*On Stresses in a Magnetostatic Field*," was read by the SECRETARY.

Quincke found that when a glass bulb containing a solution of ferric chloride was placed between the poles of a strong electromagnet, the level of the liquid, in a capillary tube attached to the bulb, fell. This has been held to require for its explanation a system of stress which differs from the magnetic stresses of electrical type. The object of this paper is to show that the experiment can be quite well explained by the stresses of electrical type.

Dr. W. WATSON gave Some Hints on the Preparation of Diagrams.

He pointed out that many curves accompanying papers submitted to scientific societies have to be re-drawn on white Bristol board before they are suitable for reproduction by photographic processes. It is now possible to obtain blue-lined section-paper such that the lines do not show in a photograph of a diagram drawn upon it. There are many advantages in using such paper. For instance, it practically dispenses with the use of scales and T-squares. When lettering diagrams a great saving of time and a uniformity of lettering can be effected by using indiarubber type. These can be purchased cheaply in different sizes and different fonts. The larger types are sold as "Easy Sign Markers," and are supplied with rods to ensure alignment and proper spacing, and also with rods by means of which the letters may be arranged upon a circular arc. A suitable ink for use with the type can be obtained by mixing Binfeld's Persian black with glycerin. Using this ink for the lettering and ordinary Indian ink for the line-work of a diagram drawn on blue-line section-paper, it is possible to slightly over expose a photograph so that the section-lines are invisible in lantern-slide prints prepared from it.

Mr. R. J. SOWTER exhibited a Portable Electroscope of high insulation and adapted to show and measure the discharging effect of radio-active substances.

Strips of gold leaf were mounted near the ends of a strip of gutta-percha tissue, leaving sufficient gutta-percha between the metal to provide the insulation. This compound strip, when doubled in two and attached to a small metal clip, constitutes a portable electroscope which may be supported on a glass rod, by the hand, or in any convenient manner. The effects of arbitrary amounts of radium, pitchblende, and uranium oxide were such as to discharge the leaves after electrification in five seconds, five minutes, and seven and a-half minutes respectively. The discharge-curves are practically straight lines. One of the electroscopes shown had leaves 9 inches long, and for large deflections was charged by an electrophorus. As an electrophorus Mr. Sowter has found a metal tea-tray supported on three glasses, together with a piece of brown paper warmed and brushed, satisfactory.

Prof. J. D. EVERETT mentioned that he had seen the discharging effect of radium shown by means of silk tassel electrified by stroking it with a rubber glove.

Mr. C. BOYS said that many years ago he had used a tea-tray and sheet of brown paper as an electrophorus, and had obtained from it sparks up to 2 inches in length.

## THE INSTITUTE OF CHEMISTRY.

THE Twenty-sixth Annual General Meeting of the Institute of Chemistry was held at 30, Bloomsbury Square, on Tuesday, March 1st, Mr. DAVID HOWARD, President, in the Chair.

The annual accounts with the Report of the Auditors having been submitted by Mr. A. Gordon Salamon, Hon. Treasurer, and duly received, Prof. TILDEN moved the adoption of the Annual Report of the Council, at the same time commenting on the general progress of the Institute. He considered it satisfactory to note that, notwithstanding the increasingly stringent regulations as to training, the very high standard of the examinations, and in spite of the loss of members by death, the number of Fellows and Associates, viz., 1098, was 251 higher than in 1894. He also commented on the scheme for the promotion of the better training of technical chemists now under the consideration of the Council.

Mr. FRISWELL seconded the report, which was then adopted.

The ballot for the election of Censors having been taken, Dr. Thomas Stevenson, Prof. J. Millar Thomson, Prof. W. A. Tilden, and Dr. J. A. Voelcker were declared elected.

The meeting proceeded to appoint Hon. Auditors, and Messrs. A. R. Ling, C. H. Cribb, and R. E. Alison were appointed.

The PRESIDENT then delivered his Address, in which he commented on the work of the Council during the past year, on the present position of the Institute, and on the work the Council at present have in hand.

He reported that the number of candidates for the examinations continued to increase, and that it had been found necessary to make arrangements for holding an extra examination in April of this year in order to avoid an excess of candidates in July. The Council, with the help of the new Treasurer, had given particular attention to the finances of the Institute; the heavy expenditure on examinations had rendered it necessary to raise the fees, but the Council had given time for students, already in course of training, to become registered under the lower fees. The alterations would not be such as to impose a great hardship on the individual candidate, and yet, by the time the new regulations were in full operation, the fees received would probably meet the cost of the examinations. Hitherto they had been conducted at a heavy loss. It had always been felt that the maintenance of the efficiency of the examinations was a primary duty of the Institute, and, in endeavouring to organise the profession—by bringing together the properly trained and competent—it had been necessary for the Institute to afford all possible facilities for this object. The principal duty of the Institute was to promote the better education and examination of public and technical analysts. Mr. Howard showed how the Institute had carried out this object in promoting the better education of professional chemists generally, and public analysts in particular. He mentioned incidentally that, from carefully collected information, he had ascertained that in England and Wales 93.5 per cent, in Scotland 89.5 per cent, and in Ireland 84.7 per cent of the appointments as public analysts, under the Sale of Food and Drugs Acts, were held by Fellows and Associates of the Institute. However no special provision had been made in respect of the training and examination of technical chemists, and it was to this matter that the Council at the present time were paying particular attention. An outline of the proposed scheme had been published in the *Proceedings*, and details will be published shortly. The Special Committee which had the matter in hand would hold a conference during the present year with manufacturers and others interested in the progress of our scientific industries. The difficulty was to bridge over the gap between the scientific training and the practical work of the technical chemist; he has to learn to think in tons instead of grammes. The

whole question would require most careful consideration, but it was hoped that the Institute might assist in overcoming the difficulty.

The Officers and Members of Council for the ensuing year were duly elected as follows:—

*President*—David Howard.

*Vice-presidents*—George Beilby; Frederick Daniel Chattaway, M.A., D.Sc.; Percy Faraday Frankland, Ph.D., F.R.S.; Edmund Albert Letts, D.Sc.; Edmund James Mills, D.Sc., F.R.S.; John Millar Thomson, LL.D., F.R.S.

*Hon. Treasurer*—Alfred Gordon Salamon, A.R.S.M.

*Members of Council*—Alfred Henry Allen; Leonard Archbutt; Edward John Bevan; Bertram Blount; Horace T. Brown, LL.D., F.R.S.; James Cameron; Alfred Chaston Chapman; Edwy Godwin Clayton; James Kear Colwell; Edward Divers, M.D., F.R.S.; James Johnstone Dobbie, M.A., D.Sc.; Bernard Dyer, D.Sc.; Thomas Fairley; Richard John Friswell; Arthur George Green; Alfred John Greenaway; Oscar Guttmann; Henry James Helm, I.S.O.; Herbert Jackson; William Walker James Nicol, M.A., D.Sc.; Frederic James Montague Page, B.Sc., A.R.S.M.; William Jackson Pope, F.R.S.; Alexander Scott, D.Sc., F.R.S.; David Alexander Sutherland; Leo Taylor; Edward William Voelcker, A.R.S.M.; Sydney Young, D.Sc. F.R.S.

## NOTICES OF BOOKS

*Transactions of the American Electro-chemical Society.* Vol. III. Third General Meeting. Published by the American Electro-chemical Society, Philadelphia, Pa. 1903. Pp. 373.

THE third general meeting of this Society was held in New York on April 16th, 17th, and 18th, 1903, under the presidency of Dr. Joseph W. Richards, when a number of interesting papers were read and discussed. Some of the most important were on the "Corrosion of Metals by Electrolysis," by A. S. Knudson, with special regard to the destructive effects of railway currents upon subterranean metals, such as water-pipes; "Ions and Electrons," by Louis A. Parsons, which will be of service in extending the new theories and hypotheses which have followed on the discovery of radium; and "Determination of Vapour Densities in an Electric Furnace," by Prof. W. Nernst.

The appendix consists of a lecture delivered to the Society on April 17th by William J. Hammer on "Radium and other Radio-active Substances, with a consideration of Phosphorescent and Fluorescent Substances; the Properties and Applications of Selenium and the Treatment of Disease by the Ultra-violet Light." In the rather wide limits of this comprehensive title, the author has endeavoured to describe certain fundamental principles connected with the phenomena upon which he has treated; and in considering these subjects, all of which may be said to be on the borderland of science, to bring out by means of experiments, lantern-slides, and illustrations, the practical and commercial side.

*The Optical Rotating Power of Organic Substances, and its Practical Applications.* By DR. HANS LANDOLT. Second Edition. Authorised English Translation, with Additions by Dr. J. H. LONG. Illustrated. Easton, Pa.: The Chemical Publishing Company. 1902. Pp. 751.

THE scope of the second edition of this work, published in 1898, and of which the present volume is a translation, is much wider than that of the first. The progress which has been made of late years in the field of optical activity rests, so far as the theoretical side is concerned, mainly upon the great interest aroused among chemists by the hypothesis of

van't Hoff and Le Bel on the relation between rotating power and the atomic structure of carbon compounds.

In a practical direction progress has been made in the improvement of apparatus and in the methods of optical analysis; while the number of optically active substances known has increased since 1879 from 300 to over 700.

The book is divided into six parts. Part I. is on the general conditions of optical activity, and includes the classification of active substances, the nature of the rotating power, the relations between rotating power and chemical constitution of carbon compounds. Part II., on the physical laws of circular polarisation, is only a short one, while Part III., on numerical values for the rotating power and specification rotation, covers many more pages.

The fourth part deals with apparatus and methods for the determination of the specific rotation, and Part V. is on the practical applications of optical rotation, such as the determination of cane-sugar, of milk-sugar, of glucose, maltose, camphor, the cinchona alkaloids, &c.

Part VI. is on the constants of rotation of active bodies, to which many additions have been made by the translator, by permission, and with the assistance, of the author.

The book is one of considerable value and importance, and will be studied to much advantage by those who have identified themselves with this particular branch of science.

*Fractional Distillation.* By SYDNEY YOUNG, D.Sc., F.R.S. With 72 Illustrations. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1903. Pp. 284.

FRACTIONAL distillation is invaluable when we wish to prepare liquids in the highest state of purity, and the eighteen years' experience of the author in researches requiring the frequent use of this manipulative method, entitles him to speak with authority on the subject. One important result of his careful investigation of the subject has been the devising of new methods and apparatus which have been described from time to time in the scientific journals.

The book is divided into nineteen chapters, and is fully illustrated. After a few preliminary chapters on more or less theoretical and elementary points, we come to Chapter VII., on "Directions for Carrying out a Fractional Distillation"; this is followed by chapters on the theoretical relations between the weight and composition of the distillate, and between the boiling-points of the residue and the distillate.

Chapters X., XI., and XII. deal with modifications of the still-head, dephlegmators, and "regulated" or "constant temperature" still-heads, and Chapter XV. is on distillation on a manufacturing scale. The application of fractional distillation to quantitative analysis is dealt with in Chapters XVI., XVII., and XVIII.; this method may be safely employed in the majority of cases for the determination of the composition of a mixture which separates normally into its components, provided that a very efficient still-head be employed and the operation be carried out very slowly. Chapter XIX. contains general remarks on the purposes for which fractional distillation is required; the interpretation of experimental results, the choice of still-head, and number of fractions, &c. The book, which is a valuable one, concludes with a short appendix on the correction of the height of the barometer for temperature, and a good comprehensive index.

*Aids to Forensic Medicine and Toxicology.* By WILLIAM MURRELL, M.D., F.R.C.P., &c. Sixth Edition, Fourteenth Thousand. London: Baillière, Tindall, and Cox. 1903. Pp. 110.

THIS book is not intended as a substitute for ordinary textbooks on forensic medicine or for lectures, but simply as an aid or reminder of facts already acquired.

Forensic medicine, or medical jurisprudence, is that portion of medical science which treats of the connection

between law and medicine, and deals with cases connected with the administration of justice, while toxicology treats of the nature and detection of poisons. These are both important branches of medical science, and the present "aid" deals with both in a clear and concise manner, and will be of service to medical men in general.

*General Information with Regard to the Gold, Diamond, and Forest Industries of British Guiana.* Edited by the Secretary of the Institute of Mines and Forests of British Guiana. Georgetown: Printed for the Government of British Guiana by Baldwin and Co. 1903. Pp. v.-25, xxiv., and xviii.

As frequent inquiries are addressed to the Colonial Office with regard to mining, &c., in British Guiana, it has been thought desirable, in the interests of the colony, to prepare and bring up to date annually a special pamphlet giving shortly the necessary information as to the gold and diamond mining industries, and the land and mining regulations, to supplement the information contained in the handbook issued by the Emigrants' Information Office. In these pages will be found information and statistics with regard to the Ballata and timber industries, as well as those of gold and diamond mining. Notes on the method of acquiring Crown lands, a digest of the mining laws of the colony, hints as to equipment, and geological notes are included, together with information as to communication with the colony, and in the colony with the gold and diamond fields.

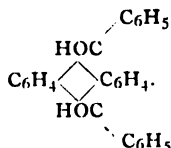
This handbook will be of service to those intending to proceed to British Guiana; the information it contains is of a varied but sound character, but it has been put together in a most inconvenient manner; though only a small volume no fewer than four systems of numbering the pages have been adopted, besides several scattered indices and appendices, the pages of which are not numbered at all. As it is proposed to issue this handbook annually, we hope to see the next edition paged in a more rational manner.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 6, February 8, 1904.

**Action of Phenylmagnesium Bromide on Anthraquinone.** Symmetric  $\gamma$ -Dihydroxyl- $\gamma$ -diphenyl Dihydride of Anthracene.—A. Haller and A. Guyot.—The authors apply M. Grignard's method to the preparation of  $\gamma$ -dihydroxyl- $\gamma$ -diphenyl dihydride of anthracene:—



**Flame Spectra of the Alkaline Metals.**—C. de Watteville.—The author examines the spectra of lithium, sodium, and potassium, and his experiments tend to verify the theory that the production of spectrum lines is purely calorific. This theory explains the fact that in the arc, where the temperature is considerably higher than that of the flame, there are more elementary zones; that is to say, more special states in which the atom is susceptible of emitting a given series of rays.

**Use of Alternating Currents for Electrolysis.**—André Brochet and Joseph Petit.—The authors make a series of interesting experiments on the solubility of copper in potassium cyanide under the influence of alternating currents.

**Formation of Phosphorus Sulphides in the Cold.**—R. Boulough.—Certain phosphorus sulphides can be produced in the cold if a little iodine is added to a solution of sulphur and of  $\text{P}_4\text{S}_3$ . The violet colour rapidly disappears, and in a day or two small crystals appear, which on analysis are shown to have the invariable composition  $\text{P}_4\text{S}_7$ .

**Action of Heat and Light on Mixtures of Phosphorus Sesquisulphide and Sulphur in Carbon Disulphide Solution.**—E. Dervin.—The author finds that heat and light act in the same manner on mixtures of sulphur and phosphorus sesquisulphide in carbon disulphide solution. A solution containing one molecule of  $\text{P}_4\text{S}_7$  to a half molecule of sulphur gives yellowish transparent needles of  $\text{P}_2\text{S}_4$ . He also ascertains that solar light completes the reaction in a month or two, whilst at a temperature of  $210^\circ$  the same effect is produced in two hours.

**Action of Carbon Dioxide on Solutions of Sodium Nitrite.**—C. Marie and R. Marquis.—The authors repeat certain very simple experiments which show decisively that carbon dioxide displaces nitrous acid in solutions of sodium nitrite.

**Constitution and Properties of Vanadium Steels.**—Léon Guillet.—The author distinguishes three distinct groups in crude vanadium steels, one of these groups being intermediate in properties between the other two. He also describes the mechanical properties of each of the groups, and shows that the steels of each present very remarkable characteristics. Lastly, he determines the fact that only vanadium steels containing less than 7 per cent of vanadium are susceptible of industrial application, on account of the fragility of those containing larger percentages of vanadium.

**Allotropic Transformations of Nickel Steels.**—O. Boudouard.—The author investigates the allotropic transformations of certain specimens of nickel steels containing 2 per cent and 30 per cent of nickel by means of curves obtained during the heating and cooling of bars of the metal.

**Diurides. Homoallantoic Ether.**—L. J. Simon.—In a previous research the author proved the existence of homoallantoic ether,  $\text{CH}_3\text{---C}(\text{NH---CO---NH}_2)_2\text{---CO}_2\text{C}_2\text{H}_5$ . This diuraminic ether, the only one known up to the present time, is obtained by the direct action of urea on ethyl pyruvate without the intervention of any condensing agent. When pure it is a white powder, micro-crystalline, and decomposed at  $200^\circ$ ; insoluble in water and most organic reagents. He also investigates certain of the chemical properties of this substance.

**Phosphoric Ethers of Glycol.**—P. Carré.—Glycol, in the same manner as glycerin, can react on the three oxyhydrides of phosphoric acid, giving rise to three ethers. The best results are obtained by acting at a temperature of  $140\text{---}145^\circ$ , and under a pressure of 15 to 18 m.m., given by a water-pump. Under these conditions the maximum etherification is attained after ten hours of heating. The mixture then contains 3.5 per cent of the triether, 42.4 per cent of the diether, and 44 per cent of the monoether, giving a total of 89.9 per cent of combined acid.

**Biochemical Synthesis of Olein and certain Ethers.**—Henri Pottevin.—In a previous investigation the author proved that oleic acid and glycerin are etherified under the influence of a pancreatic ferment, giving monolein. In consequence of the same diastatic action, he is now enabled to prepare triolein identical with natural olein.

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 7th inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. Miss L. A. Black, H. T. Davidge, H. F. Dickens, K.C., F. S. Eve, F. L. M. Forster, Miss C. E. M. Gibbons, T. G. Hull, Vivian B. Lewes, Mrs. Gerard Leigh, The Hon. Mary Portman, H. R. Prendergast, W. N. Shaw, F.R.S., J. Sorley, Dr. R. H. Scanes Spicer, The Hon. Sir Joseph Walton, and E. Wormald were elected Members.

## MISCELLANEOUS.

**Royal Institution.**—On Tuesday next, March 15, at 5 o'clock, Dr. E. A. Wallis Budge will deliver the first of two lectures on "The Doctrine of Heaven and Hell in Ancient Egypt, and the Books of the Underworld"; and on Thursday, March 17, at the same hour, Mr. Sidney Lee commences a course of two lectures on "Shakespeare as Contemporaries knew him." The Friday Evening Discourse on March 18 will be delivered by M. Henry Arthur Jones on "The Foundations of a National Drama"; and on March 25 by Professor Dewar on "Liquid Hydrogen Calorimetry."

**Relative Effects of Superphosphate and Basic Slag upon the Feeding Quality of Swedes.**—The West of Scotland Agricultural College has published, in pamphlet form, the results of a number of tests made on the above subject by Dr. John W. Paterson. The present experiment was designed to test the effects of manures upon quality by utilising the swedes grown for an accurate feeding test. One acre of roots was manured with superphosphate and one acre with basic slag, the same quantities of nitrogen and potash being added to each plot. The roots on each acre were subsequently fed to separate lots of sheep. The yield of roots from the first plot was 21 tons 0 cwt. 3 qrs., and from the second plot 19 tons 18 cwt. 0 qrs., the superphosphate producing the heavier crop. The two crops were then fed to two lots of selected sheep, 40 in each lot, the same weight of roots being given to each lot. Beside roots, a weighed and equal amount of dried grains was given daily to each lot. Hay was not considered necessary when dried grains was the concentrated food. The sheep were weighed on two dates after the commencement to find their respective gains, and it was found that while, during the first half, the two lots of sheep increased by nearly the same amount, during the second period the basic slag lot took a somewhat strong lead. Over the whole period the basic slag lot of 40 sheep made nearly 15 per cent more increase, although the sheep were otherwise similarly treated in every way. Taking the weight of crops alone, the basic slag stood to superphosphate as 94 to 100; but when actual mutton production is reckoned in, the tables are turned, and basic slag stands to superphosphate as 108 to 100. That is to say, the slag acre produced the smaller but the more valuable crop.

**The Compounds of Sulphur and Tellurium.**—A. Gutbier and F. Flury.—I. *Action of Sulphuretted Hydrogen on the Compounds of Hexavalent Tellurium.*—The authors confirm the previous results of Brauner (*Chem. Soc.*, 1895, vol. lxx., p. 545). In aqueous solution of telluric acid,  $\text{SH}_2$ , at the ordinary temperature, gives a precipitate of the composition  $\text{TeS}_3$ , but which may be looked upon as a mixture of sulphur and tellurium. In a warm solution, sulphuretted hydrogen partially reduces the  $\text{TeO}_3$  to the state of tellurous acid. In this reaction no sulphonyltelluric acid is formed. II. *Action of Sulphuretted Hydrogen on the Alkaline Solutions of Hexavalent Tellurium.*—The authors have not succeeded in isolating any sulphotellurates, which confirms the doubts expressed by Staudenmeyer (*Zeit. Anorg. Chem.*, 1895, vol. x., p. 189) on the results announced by Oppenheim (*Journ. für Prakt. Ch.*, [1], vol. lxxi., p. 270). III. *Action of Sulphuretted Hydrogen on Solutions of Tetravalent Tellurium.*—In acid solution sulphuretted hydrogen causes the precipitation of an orange-red material of the composition  $\text{TeS}_2$ . This precipitate is a mixture of S and of Te. By extracting in a Soxhlet apparatus with sulphide of carbon, the major portion of the sulphur is eliminated (the residue contains only 1.18 per cent of S). From the residue containing 1.18 per cent of sulphur, the authors have succeeded in obtaining tellurium free from sulphur, and they are continuing their researches with a view to clearing up this particular point. The paper closes with a note by M. Gutbier concerning

the criticism his previous work has received at the hands of M. Brauner (*Zeit. Anorg. Chem.*, vol. xxxi., p. 378).—*Zeit. Anorg. Chem.*, vol. xxxii., p. 272.

**Action of Phenylhydrazine on the Oxidised Compounds of Selenium and Tellurium.**—A. Gutbier.—In hydrochloric solution tellurous acid is reduced by phenylhydrazine at the ordinary temperature. In aqueous solution the same reduction takes place, with the transitory formation of an unstable bright yellow precipitate. Selenious acid dissolved in dilute alkali and treated with phenylhydrazine behaves like  $\text{TeO}_2$ . In cold alkaline solution we can obtain a pseudo-solution of selenium in alcohol, but this very unstable solution deposits selenium rapidly. The reduction of  $\text{SeO}_3$  is slower, and the author has succeeded in isolating the intermediate product,  $(\text{C}_6\text{H}_5\text{NH.NH})_2\text{SeO}_4\text{H}_2$ , in the form of small microscopic needles having a silky lustre, stable in the air, and soluble in water. This colourless solution deposits Se when raised to boiling-point in the presence of a few drops of hydrochloric acid.—*Zeit. Anorg. Chem.*, vol. xxxii., p. 257.

**A New Reaction for Aldehydes.**—E. Rimini.—It has been shown in a previous paper that the aldehydes condense with nitrohydroxylamic acid, giving the hydroxylamic acids. This reaction allows of the isolation of the latter acid with difficulty, on account of the formation of a nitrite which, by the acidulation of the solution, changes the product through the oxidising action of the oxides of nitrogen. The author uses benzene-sulphohydroxylamic acid,  $\text{C}_6\text{H}_5\text{SO}_2\text{NHOH}$ , which acts like the nitro acid but gives benzene-sulphinic acid, which does not interfere with the isolation of the hydroxylamic acid sought for. This latter is precipitated in the form of a cupric salt by the addition of acetate of copper to the aqueous acetic solution. The author has operated with formic, acetic, valeric, aminovaleric, acrylic, citral, glyoxal, glyceric, benzoic, piperonylic, anisic, salicylic, and furfural aldehydes. Formic acid, however, gives the same reaction, as it contains the aldehydic group; the author also gives the analysis of the salts of copper obtained with these different substances. He proposes to examine the application of this reaction to the qualitative and quantitative detection of the aldehydes present in alcohol and in spirits.—*Gazz. Chim. Ital.*, vol. xxxi., [2], p. 84.

**Mixed Crystals of Sulphur and Selenium.**—W. E. Ringer.—Sulphur and selenium in the fused state are miscible in all proportions; the melted mass containing a proportion of selenium greater than 10 atoms per cent crystallises with difficulty. On cooling very slowly an amorphous mass is produced. By heating them for several hours to very near their fusing-point, mixtures rich in selenium become completely crystalline. From an examination of their fusion curves, it results that all these crystalline mixtures consist of mixed crystals of sulphur and selenium; nothing points towards the formation of a double combination. These melted mixtures form three series of crystals:—1. A series of monoclinic crystals containing from 0 to 27 atoms per cent of selenium and of the same type of crystal as monoclinic sulphur. 2. A series of monoclinic crystals containing 50 to 82 atoms per cent of selenium (of the same type as the third modification of sulphur?). 3. A series of crystals formed of hexagonal rhombohedra of the type of metallic selenium, containing 87 to 100 atoms per cent. At a temperature comprised between 75 and 95.5 the crystals of the first series are transformed into orthorhombic crystals; the transformation is comparable to that of monoclinic sulphur into the orthorhombic variety. The crystals belonging to the two other series do not undergo any analogous change. At the ordinary temperature we obtain:—1. A series of rhombic crystals containing from 0 to 10 atoms per cent of selenium. 2. A series of crystals containing from 55 to 76 atoms per cent of selenium. 3. A series of crystals of the type of hexagonal selenium containing 90 to 100 atoms per cent of selenium.—*Zeit. Anorg. Chem.*, vol. xxxii., p. 183.

**Detection of Saccharine by New Reactions.**—M. Spica.—The author criticises the method of detecting saccharine by heating the product of the extraction with ether mixed with ligroin, with resorcin, and sulphuric acid. This method, though very sensitive, has the drawback of giving fluorescences with the ethereal extracts of wines and the various pharmaceutical syrups free from saccharine. This is a result of the entangling by the ether of the tannic, lactic, tartaric, malic, succinic, and other acids, capable of forming fluorescent products of condensation with resorcin. The author prefers the two following methods:—1. *Oxidation of the Saccharine into Detection of the Nitric Acid with Diphenylamine.*—He operates in a test-tube with the residue from the evaporation of the ligroin-ether which served for the extraction.  $\text{H}_2\text{SO}_4$  and a crystal of  $\text{KMnO}_4$  are added, and the tube heated gently; the  $\text{MnO}_2$  is destroyed with a few drops of oxalic solution, and after the addition of a few c.c. of hydrochlorate of diphenylamine, concentrated sulphuric acid is poured slowly in, and the surface of contact of the two liquids observed closely; a blue ring should be produced. 2. *Transformation of the Saccharine into Aminobenzoic Acid, and the Formation of an Azoic Colour.*—The residue from the extraction, containing saccharine, is treated with a few decigrams of caustic lime. Heat until slight browning occurs, treat with water, decant the aqueous solution, acidulate with  $\text{HCl}$ , and add a granule of zinc; let the reduction proceed for twenty minutes, then decant the clear liquid, and treat with a few drops of a dilute solution of  $\text{NO}_2\text{Na}$ , and finally 5 drops of  $\alpha$ -naphthylamine solution. A cherry-red colour is then observed if saccharine is present.—*Gazz. Chim. Ital.*, vol. xxxi., [2], p. 41.

**Composition of Potato Starch.**—A. Fernbach.—The author establishes the fact that potato starch is not a homogeneous substance owing to its being deposited in concentric layers, which differ in composition. He finds the chief irregularity in the composition lies in the proportion of phosphorous present in the starch.—*Comptes Rendus*, cxxxviii., No. 7.

## MEETINGS FOR THE WEEK.

**MONDAY, 14th**—Society of Arts, 8. (Cantor Lectures). "Recent Advances in Electro-chemistry," by Bertram Blount, F.I.C.

**TUESDAY, 15th.**—Royal Institution, 5. "The Doctrine of Heaven and Hell in Ancient Egypt, and the Books of the Underworld," by E. A. Wallis Budge, M.A., &c. Society of Arts, 4.30. "Recent Developments in Devonshire Lace Making," by Alan S. Cole, C.B.

**WEDNESDAY, 16th**—Society of Arts, 8. "Artificial and other Building Stones," by L. P. Ford.

Microscopical, 8. "A Note on some New Methods of Measuring the Magnifying Power of the Microscope and of Lenses generally," by Prof. A. E. Wright. Exhibition of Hand-painted Lantern Slides, illustrating Botanical Histology, prepared by A. Flatters.

Chemical, 5.30. "Mercuric Nitrite and its Decomposition by Heat," by P. C. Ray. "Note on the Higher Glycerides," by J. B. Hannay. "Nature of a Solution of Iodine in Aqueous Potassium Iodide," by C. H. Burgess and D. L. Chapman. "Reduction of 2:6-Dinitrotoluene with Hydrogen Sulphide," by J. B. Cohen and J. Marshall. "Isomeric Change of Diacylanilides into Acyl-amino-ketones—Transformation of the Dibenzoyl Toluidines into the Isomeric Methyl-benzoyl-amino-benzophenones," by F. D. Chattaway and W. H. Lewis. "Acid Esters of Methyl-substituted Succinic Acids," by W. A. Bone, J. I. Sudborough, and C. H. G. Sprankling. "Action of Ethyl  $\beta$ -iodopropionate on Ethyl Diiodoacetylenetetra-carboxylate," by O. Silberrad.

**THURSDAY, 17th.**—Royal Institution, 5. "Shakespeare as Contemporaries knew him," by Sidney Lee, Litt.D.

**FRIDAY, 18th.**—Royal Institution, 9. "The Foundations of a National Drama," by Henry Arthur Jones.

**SATURDAY, 19th.**—Royal Institution, 3. "The Life and Work of Stokes," by Right Hon. Lord Rayleigh, F.R.S.

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and Prof. E. HINTZ, Ph.D.  
Chemistry and Analysis of Foods .. .. . Prof. Dr. med. G. FRANK.  
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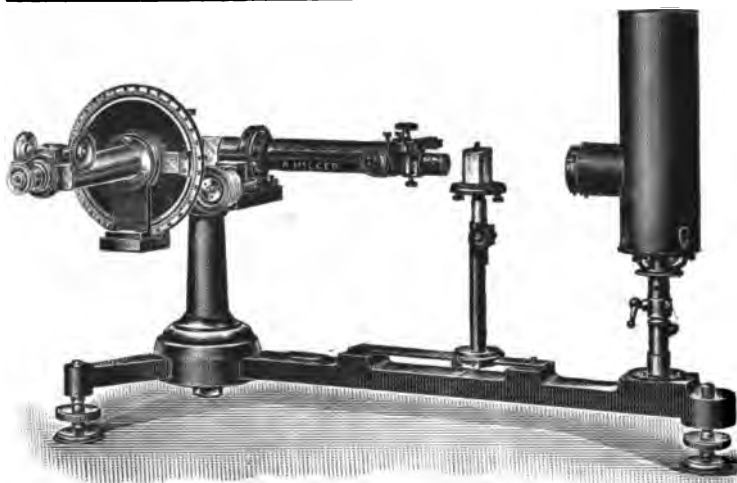
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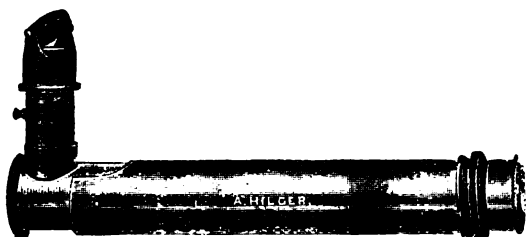
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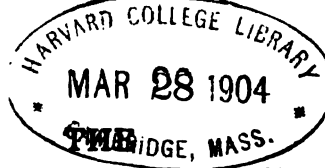
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## CONTENTS.

| ARTICLES:—                                                                                                    | PAGE |
|---------------------------------------------------------------------------------------------------------------|------|
| A Study of the Radio-activity of certain Minerals and Mineral Waters, by Hon. R. J. Strutt .....              | 133  |
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S. ....                              | 135  |
| The Life and Work of Eugène Demarçay, by A. Etard.....                                                        | 137  |
| On the Resistance of Iridio-platinum Anodes used in the Electrolysis of Alkaline Chlorides, by P. Denoe ..... | 138  |
| PROCEEDINGS OF SOCIETIES—                                                                                     |      |
| CHEMICAL SOCIETY .....                                                                                        | 140  |
| NOTICES OF BOOKS .....                                                                                        | 142  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                   | 143  |
| MISCELLANEOUS .....                                                                                           | 143  |
| MEETINGS FOR THE WEEK.....                                                                                    | 144  |

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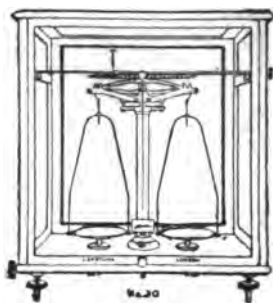
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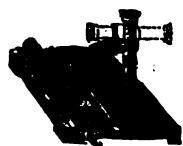


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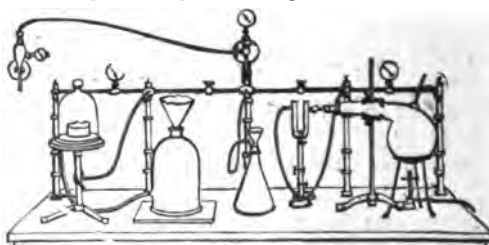
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# THE CHEMICAL NEWS.

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## A STUDY OF THE RADIO-ACTIVITY OF CERTAIN MINERALS AND MINERAL WATERS.\*

By Hon. R. J. STRUTT, Fellow of Trinity College, Cambridge.

### PART I.

A CONSIDERABLE number of minerals are known in varying degrees to be radio-active. Lists have been given by M. and Mme. Curie (*Thèse présentée à la Faculté des Sciences, Paris*, p. 19; *CHEMICAL NEWS*, lxxxviii., p. 85), and by Sir William Crookes (*Proc. Roy. Soc.*, lvi., p. 411). Except in the case of pitchblende, little has been done to determine the nature of the radio-active constituents, or to decide whether any hitherto unknown radio-active body is present.

To obtain complete information on these subjects, the only method available would be to completely analyse the mineral, and examine every precipitate and filtrate for radio-activity. This process is, of course, very tedious, and the results have to be interpreted with care, since traces of radio-active elements may often be carried down in the groups to which they do not properly belong, and thus cause confusion. A much easier method is to heat the crude mineral, and to examine the rate of decay of the emanation which it gives off. Each emanation has a characteristic time constant of decay, and by determining this we can identify it.

The method is, of course, useless for testing the presence or absence of radio-active elements, such as uranium,† which do not give off a characteristic emanation. But the great difficulty with which it may be applied to a small quantity of material, and the definiteness of the results, are great merits.

In any case when a material suspected to contain radium is obtainable in abundance, it is better to test for the presence of emanation than to look for activity in the solid. For but little of the solid material can be advantageously used in the test. Thick layers give no larger effect than thin ones, since the upper layers absorb the radiation from the lower. But the emanation can be extracted from any desired bulk of material, and the effect proportionately increased. If carbonic acid or any other gas is evolved at the same time in inconvenient quantities, it can be absorbed with a suitable reagent, and the emanation contained in it thereby concentrated.

The present paper gives the results of an examination of certain radio-active materials by this method.

No new emanation has been recognised. The results have in all cases been attributable to thorium and radium.

If any emanation decidedly more permanent than that of radium existed in the evolved gas, the method could not fail to detect it. For in every case the activity of the gas was watched until it became comparable with the very small activity due to the walls of the vessel. If a more durable emanation had been present, even in small quantities, the proportion of it present would have increased relatively to the radium emanation, and its presence would have become apparent towards the end, by a diminished rate of decay.

Small quantities of an emanation less durable than that of radium might have escaped detection; for they would have been masked by the much greater quantity of the latter.

\* A Paper read before the Royal Society, March 10, 1904.

† I have found a distinct, though feeble, emanation from re-crystallised uranium nitrate, having a rate of decay equal to that of the radium emanation. Whether this is really due to uranium, or to traces of radium, which the uranium still contains, must be left for the present an open question.

By measuring the rate of leak due to the accumulated emanation from a weighed amount, the proportion of radium present may be estimated. A comparison with the leak due to the emanation of a known weight of radium must of course be made. For this purpose it would be best to weigh out, say, a milligram of radium bromide, dissolve it in a litre of water, and evaporate a small measured quantity of the solution in a suitable tube. In this way the effect due to a standard quantity could be determined.

The method of experimenting was as follows:—

The powdered mineral was placed in a hard glass combustion tube, drawn out and sealed at one end, connected to a mercury gas-holder at the other. The mineral was heated to redness, and the gaseous products collected in the gas-holder. When the evolution of gas had ceased, the point was broken off, and air drawn into the gas-holder up to a standard volume.

For measuring the electrical effects, an electroscope was used. This was exhausted, and the gas extracted from the mineral, together with the air which had been used to make up its volume to a sufficient amount, was admitted. After a few hours, enough for the deposited activity to attain its full value, the rate of leak was read. The day and hour was noted, and the gas was pumped out into a test-tube, and stored over mercury. After a sufficient time had elapsed, it was again introduced into the apparatus by means of a syphon gas pipette,\* and the rate of leak again measured. In the meantime the apparatus had been available for making measurements with other gases.

In some cases the emanation was initially so strong that it could not be conveniently investigated. In such cases a portion of the gas was diluted with air for measuring the rate of decay at first. The concentrated material was kept until, by lapse of time, it had become weak enough to be conveniently used. Its activity was followed until it had become too small for measurement.

With this preface the results for the various minerals tried may be given in the form of a table. The rates of leak are given in scale divisions per hour. When air alone filled the apparatus, the rate of leak was 2.25 sc. div. per hour. This was in each case subtracted. (Table I.)

All the minerals give radium emanation, though in very varying quantity.

These tests were not started quickly enough to give information as to the presence of a very quickly decaying emanation. This was tested for independently.

The mineral malacone is of peculiar interest, because it has been found to contain argon as well as helium (Ramsay and Travers, *Proc. Roy. Soc.*, vol. lxiv., p. 131). Helium is formed by the degeneration of radium, and it is reasonable to assume that the other kindred gases have had a similar origin. It was hoped, therefore, that malacone might contain some new radio-active element. It is still possible that it does so, but, if so, this substance gives no emanation distinct from that of radium.

The meteorite of Augusta, Co. Virginia, has also been found to contain argon and helium. But no emanation at all could be obtained from 20 grms. of it.

The minerals were all tested for thorium emanation by drawing air over them in the cold; the only one in the above list that gives it is the Norwegian monazite, and even this does not yield it very abundantly. A crystal of thorite, however, kindly lent me by Professor Lewis, was found to give torrents of thorium emanation. Air drawn over it in the cold possesses strong discharging power. It was not permissible to heat the specimen, which might have injured it, so that the presence or absence of radium emanation in thorite could not be investigated.

There can be no doubt that the other specimens of monazite contained thorium, for they were given me by the late Mr. W. Shapleigh, who was connected with the thorium industry, and used these varieties of monazite for preparing thoria. They were, moreover, markedly radio-

\* The methods of manipulation used in storing and transferring the gases without loss were those described in Dr. Travers' book "The Study of Gases."

TABLE I.

| Mineral.         | Locality.                  | Quantity taken, in grms. | Rate of leak due to emanation (sc. div. per hour). | Rate of leak per 100 grms. | Time, in days, taken by the emanation to fall to half its initial value. |
|------------------|----------------------------|--------------------------|----------------------------------------------------|----------------------------|--------------------------------------------------------------------------|
| Samarskite . .   | North Carolina, U.S.A. . . | 20                       | 20,600                                             | 103,000                    | 3'48                                                                     |
| Fergusonite . .  | Norway? . . . . .          | 7                        | 4,280                                              | 61,000                     | 3'80                                                                     |
| Pitchblende . .  | Cornwall . . . . .         | 40                       | 11,900                                             | 29,800                     | 3'50                                                                     |
| Malacone . .     | Hitteroe, Norway . . . .   | 20                       | 1,440                                              | 7,200                      | 3'81                                                                     |
| Monazite . . . . | Norway . . . . .           | 51                       | 2,060                                              | 4,000                      | 3'50                                                                     |
|                  | North Carolina . . . . .   | 82                       | 37                                                 | 45                         | 3'81                                                                     |
|                  | Brazil . . . . .           | 54                       | 11                                                 | 24                         | 3'80                                                                     |
| Zircon . . . .   | North Carolina . . . . .   | 60                       | 24'6                                               | 41                         | 4'05                                                                     |

TABLE II.

| Material.                                 | Quantity taken, in grms.              | Rate of leak due to emanation (sc. div. per hour). | Rate of leak due to emanation from 100 grms. | Time, in days, taken by the emanation to fall to half its initial value. |
|-------------------------------------------|---------------------------------------|----------------------------------------------------|----------------------------------------------|--------------------------------------------------------------------------|
| King's Spring, Bath . .                   | { Deposit from inside of well . . . . | 10                                                 | 250                                          | 3'60                                                                     |
|                                           | { Deposit from tank . . . . .         | 12                                                 | 78'2                                         | —                                                                        |
|                                           | { Saline residue from water . . . .   | 18                                                 | 12'4                                         | —                                                                        |
| Old Royal Spring, Bath . .                | { Deposit from channel near well . .  | 10                                                 | 63'5                                         | —                                                                        |
|                                           | { Deposit from bottom of tank . . .   | 15                                                 | 60                                           | —                                                                        |
| Hard deposit from sides of tank . . . . . | 25                                    | 43                                                 | 173                                          | 3'58                                                                     |
| Buxton deposit . . . . .                  | 26                                    | 356                                                | 1370                                         | 3'81                                                                     |

active, while the amount of radium emanation obtained from them was so small that their activity could not be mainly due to radium. They probably contain the thorium in what Rutherford and Soddy call the de-emanated condition. That is, the thorium emanation, though formed, is not able to escape.

It is a remarkable fact that these varieties of monazite, though they contain practically no radium, yield helium in fair quantity. There are several explanations possible. The radium originally present may have almost completely decayed into helium, and any other products which it may yield; or it may be that thorium, as well as radium, yields helium by its decomposition; or, lastly, the helium may not, in this instance, have been generated by radio-active changes at all.

It is interesting to know whether the minerals retain all the radium emanation which they generate when heat is not used to expel it. Two cases were examined. 114 grms. of powdered samarskite were kept for three weeks in a sealed glass tube. The air was pumped out and tested. It was found to contain about  $1/150$  part of the emanation, which could have been extracted by heat.

A similar experiment with malacone showed that about one-fiftieth of its emanation was able to escape in the cold.

It appears therefore that these minerals retain nearly all their emanation. The same is probably true of the helium produced by the emanation. Samarskite which had been heated to redness was found to retain its emanation in the cold about as well as before.

#### PART II.

I happened to possess a small sample of a red deposit, coloured by iron, which is left by the water of the King's Spring, at Bath. It occurred to me that it might be worth while to test this for radio-activity. The result was to show that the deposit was markedly active. On leaving it in the testing vessel (which was closed airtight) for a few days, the activity was found to increase to several times its initial value. This shows that the deposit gives off an emanation freely, even without heat.

Experiments were then made to test the rate of decay of this emanation. It proved to be identical with the rate of decay of the emanation of radium.\* The activity is wholly due to that element.

\* In the first experiment made, I obtained a small residual leak when the radium emanation had decayed. This was attributed to a new emanation, of greater durability. But I have failed to repeat the experiment, and am forced to conclude that the leak was due to a failure of the quartz insulation, owing to the presence of moisture. It is very difficult to understand how this can have happened, for the gas was passed through drying tubes. When the rate of leak was tested with air in the apparatus, it had always a perfectly definite and constant small value.

This deposit was collected inside the King's Well itself, where the hot water issues from the ground. Other deposits are left in the tanks and pipes. They are less active than that collected near the source.

Deposits from another of the hot springs at Bath, that known as the Old Royal Spring, have also been tested. These were found to be active also. In this case there was no opportunity of collecting the deposit at the well-head itself, but it was found that the deposit left in the channel near the source was more active than that in the tanks further from it.

It was interesting to determine whether the water itself contained any radium in solution. There could be little doubt that there must be traces left in solution, after the deposit had settled out. But since the Bath water contains abundance of sulphates, and since radium sulphate is one of the most insoluble salts known, there could not be more than the merest traces present. The sulphate of barium is very much less soluble than that of strontium. And presumably the sulphate of radium is much less soluble still. Barium sulphate requires half a million times its weight of water to dissolve it; radium sulphate perhaps several hundred million times its own weight.

About 10 litres of the Bath water were evaporated to dryness. The resulting saline residue was sealed up in a hard glass tube, and left for about a fortnight to generate a stock of emanation. On heating, a distinct emanation was obtained, giving several times the rate of leak that air did. A deposit, similar to that from the Bath water, but black in colour, can be collected from the source of the hot springs of Buxton. It has been analysed by Dr. J. C. Thresh (*Proc. Chem. Soc.*, January 17, 1882), and I am indebted to his kindness for a specimen of it. This deposit was found to contain radium also; the proportion present being not very different from what was found in the case of some of the Bath deposits.

Table II. gives the quantitative data for these emanations from these deposits. The rates of leak are on the same scale as those in Table I.

It will be seen that the richest of the deposits is some thirty-six times more active than the salt obtained by evaporating the water.

Although the agreement in the rate of decay of the emanation seemed sufficient to prove that the activity was really due to radium, yet it was thought desirable to show that the chemical properties of the active constituent were in agreement with this conclusion. 200 grms. of the richest deposit were treated with dilute sulphuric acid. The activity was all in the insoluble residue, which was dirty white in colour, and amounted to about half of the entire quantity of deposit. The residue was boiled with strong sodium

carbonate solution. This was washed away, and the mass extracted with hydrochloric acid. The hydrochloric acid solution gave a slight precipitate with sulphuric acid. This precipitate was collected, and found to be strongly active, so that there is every reason to conclude that the activity of the deposit is due to the presence of radium.

The presence of radium in the Bath water and deposits is of special interest because of the occurrence of helium in the gas which rises with the spring (Rayleigh, *Proc. Roy. Soc.*, vol. ix., p. 56). There can be little doubt that the helium owes its origin to the same store of radium that supplies the water.

It is interesting to estimate the quantity of radium annually delivered by the spring. Part of this is in the deposit; part in the water. But the annual yield of deposit does not exceed a few hundred-weight at the most. And although it is much richer in radium than the dissolved salt, the quantity of the latter is so enormously greater, that the deposit may be neglected. According to the estimate of Sir A. C. Ramsay, the late Director of the Geological Survey, the salt annually delivered by the spring would be equivalent in volume to a column 9 feet in diameter and 140 feet high. Taking the density to be twice that of water, this would weigh about 500,000 kilograms.

Now the saline residue gives about 1/1500 part of the quantity of emanation that samarskite gives. Let us assume that the latter contains one-millionth part of radium, which is, I think, an outside estimate. At that rate, the annual delivery of radium by the spring amounts to about one-third grm. The volume of gas which the spring delivered is about 100 cubic feet per day (Williamson, *Brit. Assoc. Reports*, 1865, p. 380). About 1/1000 part of this is helium, so that about 3 litres of helium is given off daily, or about 1000 litres per annum. The proportion of helium to radium thus indicated is of the same order as in the radio-active minerals, though somewhat larger. This is in accordance with the view that the spring draws its supplies from the disintegration of such minerals. In obtaining the various materials from the Bath springs, I have had the great advantage of Mr. Sydenham's help. His knowledge of everything connected with the springs has been of great assistance.

In addition to the Bath and Buxton waters I have examined several others.

A sample of the Cheltenham saline water, and also a deposit left in the pipes, was kindly sent me by Mr. G. Ballinger. But no emanation could be obtained, either from the dissolved salts or from the deposit.

The boiler crust from a domestic hot-water pipe, Terling, Essex, was examined, but the result was again negative.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.\*

By CHARLES BASKERVILLE, Ph.D., F.C.S.,  
Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 123).

I HAVE referred somewhat in detail elsewhere to the factors producing variations in the absorption, as well as the advantages and disadvantages of the phosphorescent and reversal spectra ("The Rare Earth Crusade," *loc. cit.*).

Without doubt the spectroscopic criteria are the most valuable we have in judging finally the elements, and mayhap will remain so; but in my humble opinion, such have not alone sufficient authority, as yet, to usher the aspirant to a place among the elect. The contention frames itself, however, in an expression of the need for uniformity.

Whether we follow the most advanced metaphysico-chemical teachings or no, if there be any one concept upon which modern practical chemical thought depends, it is the law of definiteness of composition. There may be, and doubtless are, definite, perhaps invariable, properties of our elements other than their combining proportions, the atomic weights, if you please; yet, as far as we know, they approximate more closely than any fixed, if not permanent, ratios. Many of these values, by which we lay such store, are dependent upon data\* in which, I venture the assertion, too great confidence has been bestowed, or opinions to which sufficient attention has not been given.

Although in this connection we shall give little heed to the suggested variability of the relative values, it may be remarked that Boutlerow, noting the variations observed in 1880 by Schützenberger, who, by the use of different atomic weights, obtained analyses summing 101 instead of 100, expressed the opinion that the chemical value of a constant weight, or rather mass of an element, may vary; that the so-called atomic weight of an element may be simply the carrier of a certain amount of chemical energy which is variable within narrow limits (see also Crookes). Wurtz's summary of Boutlerow's views, at a meeting of the Chemical Society of Paris, provoked an interesting discussion. Cooke later published a statement that he had expressed similar views more than twenty-five years before. That is, in 1855, he had questioned the absolute character of the law of definite proportions, and had suggested that the variability was occasioned by the very weak affinity between elements manifesting a fluctuating composition. Without doubt "The Possible Significance of Changing Atomic Volume," in which a suggestion as to the probable source of the heat of chemical combination is put forward by T. W. Richards, bears directly upon this phase of the problem (*Proc. Am. Acad. Arts and Sciences*, 1901, xxvii., 1, and 1902, xxvii., 399; *CHEMICAL NEWS*, lxxvi., 81).

While the atomic mass values depend directly upon the ratio between the constituents of the compounds, they rest equally upon the molecular weights. Many of the latter attributed to salts of some of the rare earths depend solely upon the specific (*Berichte*, 1880, xiii., 1461) heat determinations of Hillebrand and Norton (*Pogg. Ann.*, 156 *et seq.*), Nilson and Pettersson (*Berichte*, 1880, lxiii., 146), who, in the light of subsequent investigations we know, worked with complexes. To be sure, those elements which were apparently exceptions to the law of Dulong and Petit possess low atomic weights (glucinum, boron, carbon, silicon, aluminum, and sulphur), and have for the most part been brought into harmony. "The specific heats of all substances vary with the temperature at which they are measured; and though the variation is often slight, it is occasionally of relatively great dimensions. When this is so in the case of an element, the question arises:—At what temperature must the measurement of the specific heat be made in order to get numbers comparable with those of the other elements? No definite answer has been given to this question, but it is found that as the temperature rises the specific heat seems to approach a limiting value, and this value is not in general far removed from that which would make the atomic heat approximately equal 6.4 ("Introduction to Physical Chemistry," James Walker, London, p. 33). In view of this allotropism, and the work of Richards adverted to, it appears that a revision of the specific heat values now taken is necessary before we can accept fully this law, which has been most helpful.

Time will not admit of detailed statements, and it is unnecessary in this presence to more than call attention to the fact that what has been said is not applicable to each specific case. "La critique est facile, mais l'art est difficile," as Berthelot ("Les Origines de l'Alchimie," Paris, 1885) has said, yet we must appreciate that all our laws have their limitations. "Man being servant and interpreter of nature can do and understand so much, and so much only, as he has observed in fact or in thought in the course

\* Address of the Vice-President of Section C (Chemistry) of the American Association for the Advancement of Science, St. Louis Meeting, December 28, 1903.

\* Others have been referred to in the Address to which this a sequel (*loc. cit.*).

of nature. Beyond this he neither knows anything nor can do anything" (Bacon's "Novum Organum," Aphorism I.).

A glance at the extensive, even censored, list of claimants will evoke serious thought. "Thus was the building left ridiculous" (Milton, "Tower of Babel"). The difficulties briefly outlined and the causes for lack in uniformity are by no means insurmountable, but will continue until more systematic direction and prosecution of the work come about. Investigators in pure chemistry as a rule hold professorships, or other positions making equal demand upon their time. Furthermore, it is extremely rare that one man can become a master of the various delicate operations hinted at. Mallet (Stas Memorial Lecture, Chemical Society, London, delivered December 13, 1892) made a proposition for systematising atomic weight work, and F. W. Clarke in this country (Presidential Address before the American Chemical Society) and abroad (Wilde Lecture at the Dalton Centenary, Manchester, 1903) has urged the establishment of an institute for its prosecution. This appeals to all interested in what we are pleased to term the exact sciences, and doubtless in time will come about. For the time being, however, it is not unreasonable to suppose that a concerted appeal of the chemists of this country to the direction of the munificent endowment recently made American science for funds to clarify the elemental enigma presented above would be in vain. There are splendidly equipped chemical departments in some of our great American universities which would make room for, and cordially welcome, I am sure, a selected corps of supported researchers, who would test the claims of each of these and other elemental aspirants. Such a community of effort should receive even greater substantial assistance from governments and corporations than has been accorded individuals. I need only refer to the aid given the Curies by the Austrian government, and generosity shown by the Welsbach Lighting Company in this country to several investigators, especially myself.

It must be evident to all that we are not indulging in special pleading, for every phase of that division of science designated chemistry rests upon what we choose to term the elements.

Victor Meyer, referring to the phantasies of science, said (lecture on "The Chemical Problems of To-day" before the Association of German Naturalists and Physicians at Heidelberg, September, 1889; *CHEMICAL NEWS*, lxi., 21):—"He, however, who only knows chemistry as a tradition of perfectly clear facts, or who thinks to see the real soul of chemical study in measuring physical phenomena which accompany chemical transformations, feels no breath of this enjoyment." Reflecting upon the good and ill that have come to us through unrestrained imagination, we may give a careful acceptance of Newton's "physics, beware of metaphysics," for as Clifford wrote, "Doubtless there shall, by and by, be laws as far transcending those we know as they do the simplest observations."

The graphic representation of the elements, "the foundation stones of the material universe which, amid the wreck of composite matter, remained unbroken and unworn," as Maxwell gracefully spoke of them, has often been mistaken for the periodic law. Carnelley's "reasonable explanations" of the periodic law were given a respectful hearing and forgotten.\*

"Granting that the chemical characteristics of an element are connected with its atomic weight, we have, however, no right to assume them to be dependent upon that fact alone" (Living). Hinrichs ("Atom Mechanics," vol. i., p. 242, St. Louis, 1894) says weight and form, concerning the latter of which I am ignorant. No doubt the pendulum lately has swung back toward Berzelian thought revived by the like masters, van 't Hoff and Arrhenius.

\* He regarded the elements as compounds of carbon and other analogous to the hydrocarbon radicals, and suggested that all known bodies are made up of three primary elements—carbon, hydrogen, and ether—truly an assumption which cannot be disproved. Aberdeen Meeting, British Association.

Le Verrier predicted the planet Neptune, and his predictions were verified. While all of Mendeleeff's predictions, specific and tacit, have not been verified, some have. Ramsay (Address before the Chemical Section, British Association, Toronto Meeting, 1898) and others, without a periodic guide, predicted certain of the inert gases, which predictions have been verified.

Victor Meyer, in speaking of the completion of the Mendeleeff table, calls attention to the summing up of one hundred elements, from which it appears that 258 would be the limit to our atomic mass equivalents. I am not prepared positively to contradict such a conclusion at the present time, but there are reasons for thinking otherwise.

Clarke has shown that the mean density of the earth, 5.5 to 5.6, is more than double that of the rocky crust, and "the difference may be accounted for as a result of pressure, or by supposing that, as the globe cooled, the heavier elements accumulated towards the centre" ("The Relative Abundance of the Chemical Elements," F. W. Clarke, read before the Philosophical Society of Washington, October 26, 1899; *CHEMICAL NEWS*, lxii., 31). While it is quite impossible to judge of the order of this intramundane pressure, I am not aware of such marked changes being brought about in the specific gravities of the heavier solid elements or their compounds either by pressure, allotropic or isomeric changes, except the cerebral argentaurum of the late S. H. Emmens (*Argentaurum Papers* published by Emmens, New York). The examinations of volcanic dusts by Hartley (Royal Society, February 21, 1901; *CHEMICAL NEWS*, lxxxiii., 174), Fleet (*Abstr. Proc. Geol. Soc.*, 1902, 117; *Journ. Chem. Soc.*, 1902, lxxxi.—lxxxii., [2], 518) and others appear to contradict the latter explanation, although we are unable to state the depth, perhaps within the shell considered by Clarke, at which volcanoes begin their boisterous activity. While awaiting a fulfilment of Martinez' ("La Nature," *Sc. Am. Sup.*, 1886, xxi., 546) project to explore the earth's centre, we may offer a third solution, not wholly unscientific, as it can do no harm, and has nought to do with any yellow peril in science, namely, the existence of elements with atomic weights higher than those set by the silent limit of periodic tables.

"Most molecules—probably all—are wrecked by intense heat, or, in other words, by intense vibratory motion, and many are wrecked by a very impure heat of the proper quality. Indeed, a weak force, which bears a considerable relation to the construction of the molecule, can by timely savings and accumulation accomplish what a strong force out of relation fails to achieve" (Tyndall in *Longman's Magazine*).

As hinted at in the earlier portion of this unconsciously prolonged address, many have theorised as to the ultimate composition of matter. The logic of Larmor's theory (*Phil. Mag.*, 1897, 506), involving the idea of an ionic substratum of matter, the support of J. J. Thomson's experiments (*Phil. Mag.*, October, 1897, 312), the confirmation of Zeeman's phenomenon, the emanations of Rutherford, Martin's explanations (*CHEMICAL NEWS*, 1902, lxxxv., 205), cannot fail to cause credence in the correctness of Crookes's idea of a fourth state of Matter (*Phil. Trans.*, 1881, ii., 433). In the inaugural address as President of the British Association, 1898, he acknowledges in the mechanical construction of the Röntgen ray tubes a suggestion by Silvanus Thompson to use for the anti-cathode a metal of high atomic weight. Osmium and iridium were used, thorium tried, and in 1896 Crookes obtained better results with metallic uranium than platinum.

These and the facts that most of the elements with high atomic weights, in fact all above 200 (thallium not reported on), exhibit radio-active properties, are doubtless closely associated, and have to do with the eventual composition of matter. I have unverified observations which go to show the existence of at least one element with a very high atomic weight. If it be confirmed, then we have them now or they are making, and probably breaking up, as shown by that marvellous class of elements in the discovery of which the Curies have been pioneers.



(See the exquisite paper by M<sup>de</sup>. Curie on "Radio-active Substances," CHEMICAL NEWS, vol. lxxxviii., 85 *et seq.*; also "Radio-active Lead," Hofmann and Straus, *Berichte*, xxxiv., 3033; Pellini, *loc. cit.*, on "Radio-active Tellurium"; Strutt, *Phil. Mag.*, vi., 113, Elster and Geitel, Giesel, Marckwald, &c.).

If our ideas that all known elements come from some primordial material be true, then it stands to reason that we are coming in time perhaps to that fixed thing, a frozen ether, the fifth state of matter. I may make use of dangerous analogy and liken our known elements, arranged in a perfected, natural system, as the visible material spectrum, while electrons, &c., constitute the ultra-violet and cosmyle composes the infra-red, either one of the latter by proper conditions being convertible into perceptible elemental matter. No positive evidence supports these ideas, but I like to fancy scientific endeavour as the sea—calm and serene, supporting and mirroring that which is below it, bearing that which is upon it, reaching to and reflecting that which is above it, moving all the while; yet, torn and rent at times by conflict from without and contest within, it runs; it beats against the shores of the unknown, making rapid progress here, meeting stubborn resistance there, compassing it, to destroy but to rebuild elsewhere; and the existence of those within it! "Like that of Paul, our life should be a consecrated unrest."

(To be continued).

## THE LIFE AND WORK OF EUGÈNE DEMARCAÏ.

By A. ETARD.

In all ages, men capable of acquiring personal views on the philosophy of science have been rare. Among them was Demarcay, whose recent death we have to deplore.

Born on January 1st, 1852, he studied at the Lycée Condorcet, then a year of liberty in England freed him from childish restraint and prepared him for vigorous manhood.

At the age of eighteen he entered the Ecole Polytechnique, in 1870, at a time of trouble which quickly brought young spirits to maturity; he soon left and returned to the same laboratories as assistant under Cahours.

The upright spirit, the free independence tempered with good humour, and a good education, soon brought Demarcay the friendship of the professors of his time. They were Cahours, Würtz, Deville, Dumas, Friedel, Cornu, Schutzenberger, and Lecoq du Boisbaubran.

Early drawn towards pure science, he became associated with several of his contemporaries who were of the same turn of mind: Moissan, Gautier, Becquerel, Leauté, Olivier, Curie—the younger men of the time.

After some time he gave up his post at the Ecole Polytechnique so as to pursue his researches in the laboratory without interruption. But before settling down he made a journey, through Algeria, Egypt, and India to further extend his views of life. Being a good naturalist he was able to appreciate, over large tracts of country, the succession of living creatures. Equally enamoured of geology and the study of languages, these journeys enabled him to observe and to compare—a sure means of escaping from the hard and fast ritual of our classical knowledge, and to approach science with a wider ideal.

The first research of Demarcay's was in organic chemistry. In 1876 he commenced the examination of essence of anthesis and of acetylacetic ether. These studies of organic chemistry absorbed all his attention, and gave surprising results.

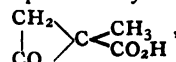
The study of essence of Roman camomile was one of the first works carried out on the complexity of odorous essences produced by vegetables. From this natural oil Demarcay isolated the  $C_5$  terpenes and acid ethers belonging to the non-saturated series. The idea of this

research was at the time nothing more than a scientific investigation; in our days it has been developed and applied by many workers in the perfume industry.

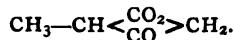
Acetylacetic ether had already, in 1879, given birth to a large number of derivatives, and was looked upon as a synthetic agent.

Demarcay was not long in producing, by the help of this substance, a series of new crystalline acids of such a curious nature that all controversy as to their constitution is not yet finished; this was the tetric series.

In 1878, Demarcay described the first body of this series,  $(C_4H_4O_2)_3H_2O$ . Later, Pawlow thought he recognised in it a  $C_5$  body,  $CH_3-CO-C=CH-CO_2H$ ; then came another conception represented by—



which is not yet fixed, for in 1902 a ring in  $C_4$  re-appeared in the formula—



There are no true successives in these matters; the critic may err, and further researches are necessary to resolve the problem set in 1879 on the tetric series. The various interpretations show that it is a deep problem.

In another group of researches, undertaken in collaboration with M. Cahours, Demarcay increased our knowledge of the stanethyils, the etherification of the primary, secondary, and tertiary alcohols by oxalic acid, and the acids from  $C_7$  to  $C_{10}$ , which accompany the distillation of the stearine acids in superheated steam.

Well known by his works and writings on organic chemistry, and especially by his demonstration of the aromatic cycle in Würtz's Dictionary, and presented in 1879 by the Chemical Section of the Academy of Science to the seat left vacant by the death of Regnault, Demarcay might well expect to have twenty or even thirty years before him. He was desirous of studying physical nature in the widest sense, not along the broad successful paths, but in the narrow ways confined to the few. After various attempts in different directions Demarcay became the man as he was known to his latest readers—the man of rare earths and of spectra.

At the commencement of this evolution the young savant attempted to constitute a complex chemistry of nitrogen and of sulphur.

After the publication of his first researches on the sulphides of nitrogen, he was the victim of a serious accident. An explosion shattered a cast-iron vessel, and he had an eye completely destroyed. When he recovered from the shock, and though more exposed to the risk of accident than before, Demarcay set himself to study compressed gases from the point of view of the action that the cold produced by expansion might exercise on the power of combination of the elements.

During 1881 and 1882, he had constructed in his laboratory in the Boulevard Berthier, an apparatus with three concentric vessels 1.5 metres high and 0.75 metre in diameter. A six horse-power gas-engine drove the compression pumps, and the temperature fell rapidly in the central portion of the apparatus to 85° below zero. It would require the resources of a factory and a large outlay of capital to obtain better returns than from this freezing machine. It is to be regretted that the plans were never published, as they anticipated the tubular apparatus now in use. I think with regret of the disappearance of a piece of apparatus that would now hold such an interesting place in the history of the liquefaction of gases. Though it could be foreseen already that it would be possible to approach very close to absolute zero, Demarcay gave up the research that he could only attempt with means that appeared to him to be too feeble. Seeking always for the essential relations that might exist between physical forces and chemical material, he organised the finest apparatus for producing vacua in Paris. By means of various

systems of mercury-falls, the vacuum became sufficiently good for the carrying out of conclusive experiments. In 1883, an interesting summary of observations appeared on the volatility of zinc, cadmium, and gold in the cold. It was shown that matters which appeared fixed to us is slowly dissipated *in vacuo*, and without doubt, though with diminished speed, they are all volatile at the ordinary atmospheric pressure.

Following up his inquiries on physical questions, Demarçay proposed to examine the variations of the spark spectra at the highest temperatures then attainable. With this object he constructed a coil with a short secondary wire of large diameter, giving very intense, globular, luminous, and hot sparks. These sparks have all the characteristics in spectroscopic practice of a small arc passing between poles of pure metals of different kinds, or, in the case of saline solutions, between two poles of pure platinum. Such an arrangement limits the lines foreign to the substance under examination to the known spectrum of the pure platinum wire serving as the electrodes.

It is with this apparatus that M. Demarçay instituted the method of separation of the rare earths. In 1900, the Parisian firm of Chenal and Douillet exhibited a collection of enormous crystals of pure salts of neodymium, praseodymium, samarium, &c., obtained by closely following Demarçay's instructions, and this caused considerable astonishment among certain specialist chemists throughout the world, who had written much on the subject, and some of whom had exhibited pasty masses devoid of any well defined colour.

In the separation of the very closely allied rare earths, Demarçay practised fractionation like all the other chemists, but he knew how to do it. The following is a brief summary of his method:—The salts of the numerous rare earths are fractionated in aqueous solution, and it matters little what salt is used, so long as it is relatively slightly soluble, as is the case with the double salts. At first the separations proceed, and we have every reason to believe that the only good method has been found. This state of affairs must be utilised to isolate that portion which goes best. Soon the best conducted work shows no further progress. It is no use revolting against nature; the work is stopped; spectrum analysis has shown that traces of salts, other than those we wish to separate, have accumulated in the fractions which no longer advanced; these are calcium and yttrium. Everything is stopped until these interfering elements have been got rid of by known means; then we again make the double salt on which the fractionation in aqueous solution had been previously carried out as if nothing had intervened, and the separation again gives good results. However, a time comes, in spite of everything, when the separation ceases, a state of equilibrium has been produced which cannot be upset. The salts of the rare earths, and the accompanying salt and the water, have become arranged naturally into unchangeable proportions. It is necessary to destroy this equilibrium, either by continuing the fractionation in aqueous solution but changing the accompanying salt, or by changing the nature of the solvent. It was in this latter manner that Demarçay, whose method I have only described summarily, often fractionated in fuming nitric acid instead of water, making use of nitrate of magnesium as the adjuvant salt. He also used with advantage a medium composed of the double sulphates of the rare earths with rubidium.

In this kind of work, too briefly described, our lamented *confrère* knew how to seize the right moment, and in this he was an artist in science. In all that is most elevated science teaches manipulation to its workers through the conception of philosophy and the vision of art.

Proceeding on this idea, Demarçay himself only undertook the most difficult fractionations. He succeeded in discovering an element most extremely rare in nature, europium, and would soon have fixed the individuality of this principal body which figures in what we call "erbium," if death had not removed him.

The laboratory where Demarçay worked was one of great renown. There, and there only, a complicated spectrum was read like an open book. Everyone who thought he had found a new pure element took it to Demarçay and was immediately enlightened. One of our eminent physicists, M. Curie, had a parcel of salts of barium examined in this manner at the commencement of his research on radium, and in which M. Demarçay immediately observed a line he did not recognise.

To-day, savants feel keenly the void left by the only man who could inform them with certainty as to the nature and purity of an element, or on the neglected residues of a new mineral.

Demarçay foresaw his own approaching death, but was conscious of a duty performed, and grateful for the years he had lived. He asked for no further reward than that felt by a keen intelligence when it gives rise to a flash of thought that will be remembered throughout the world.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 4.

#### ON THE RESISTANCE OF IRIDIO-PLATINUM ANODES USED IN THE ELECTROLYSIS OF ALKALINE CHLORIDES.

By P. DENSO.

HABER (*Zeit. für Anorg. Chem.*, 1898, vol. xvi., p. 438) effected the electrolysis of hydrochloric acid of different concentrations and at different temperatures with electrodes of platinum and iridio-platinum, and he found that the anodes containing from 10 to 25 per cent of iridium were not attacked.

It became of interest to see if anodes in the form of very thin sheets could be employed with advantage in the electrolysis of the alkaline chlorides. With electrodes of such thinness the cost would be considerably reduced, and if they were sufficiently resisting they might, in certain cases, replace carbon electrodes. The results of the experiments agree with those obtained by Haber.

The experiments were carried out by means of a series of electrodes of iridio-platinum of two different sizes, and containing from 7.6 to 15 per cent of iridium. They had a thickness of only 0.007 m.m., and were soldered to a wire of the same metal 1 m.m. in diameter. The large electrodes were 40 m.m. wide and 125 m.m. long, and consequently had a total surface area of 1 square decimetre; the small electrodes were 20 m.m. wide and 125 m.m. long, and had an area of 0.5 square decimetre.

The electrodes described above were used for the electrolysis of alkaline chlorides under various conditions; for instance:—

The electrolysis of a concentrated solution of chloride of potassium at 20° without a diaphragm.

The electrolysis of a concentrated solution of chloride of potassium at 80° without a diaphragm. (These solutions of chloride of potassium were treated with 2 per cent of chromate of potassium).

The electrolysis of a concentrated solution of chloride of sodium at 20° with a diaphragm.

The electrolysis of a concentrated solution of chloride of sodium at 80° with a diaphragm.

In the electrolysis of solutions of chloride of sodium, the anodes were placed in a large volume of liquid, about 7 litres.

After these experiments were finished we passed to the electrolysis of hydrochloric acid at 20 per cent at a temperature of 80°, as the chlorine given off by hydrochloric acid has a more energetic action on iridio-platinum than that given off by solutions of the chlorides. In these experiments the anode under examination was placed between two cathodes.

The results of all these experiments are given in the accompanying table.

| No. | Electrode.                                    | Electrolyte.                                          | Current.             | Weights of electrodes.                                   |                                      |                                                                      |
|-----|-----------------------------------------------|-------------------------------------------------------|----------------------|----------------------------------------------------------|--------------------------------------|----------------------------------------------------------------------|
|     |                                               |                                                       |                      |                                                          | Grms.                                | Grm.                                                                 |
| 1.  | 10 per cent iridium.<br>O = 1 sq. decim.      | Chloride of potassium, without diaphragm. T = 20—50°. | I = 10.5<br>d = 10.5 | At first . . . .<br>After 800 a.-h.                      | 4.8745<br>4.8744                     | Diminution . . . . 0.0001                                            |
| 2.  | 10 per cent iridium.<br>O = 1 sq. decim.      | Chloride of potassium, without diaphragm. T = 80°.    | I = 30<br>d = 30     | At first . . . .<br>After 1200 a.-h.                     | 6.7705<br>6.7700                     | Diminution . . . . 0.0005                                            |
| 3.  | 10 per cent iridium.<br>O = 1 sq. decim.      | Chloride of sodium, with diaphragm. T = 20°.          | I = 10<br>d = 10     | At first . . . .<br>After 240 a.-h.                      | 8.4525<br>8.4525                     | Diminution . . . . 0.0000                                            |
| 4.  | 9.5 per cent iridium.<br>O = 1.2 sq. decim.   | Chloride of sodium, with diaphragm. T = 80°.          | I = 20<br>d = 16.7   | At first . . . .<br>After 200 a.-h.<br>" 400 "           | 9.8910<br>9.8875<br>9.8871           | Diminution . . . . 0.0035<br>Further diminution 0.0004               |
| 5.  | 7.63 per cent iridium.<br>O = 0.5 sq. decim.  | HCl, 20 per cent. T = 80°.                            | I = 10<br>d = 20     | At first . . . .<br>After 70 a.-h.<br>" 140 "<br>" 210 " | 5.4061<br>5.4051<br>5.4044<br>5.4044 | Diminution . . . . 0.0010<br>Further diminution 0.0007<br>" " 0.0000 |
| 6.  | 9.5 per cent iridium.<br>O = 0.5 sq. decim.   | HCl, 20 per cent. T = 80°.                            | I = 5<br>d = 10      | At first . . . .<br>After 10 a.-h.<br>" 50 "<br>" 80 "   | 5.6588<br>5.6559<br>5.6552<br>5.6550 | Diminution . . . . 0.0029<br>Further diminution 0.0007<br>" " 0.0002 |
| 7.  | 9.5 per cent iridium.<br>O = 0.5 sq. decim.   | HCl, 20 per cent. T = 80°.                            | I = 10<br>d = 20     | At first . . . .<br>After 10 a.-h.<br>" 35 "<br>" 90 "   | 5.6620<br>5.6620<br>5.6620<br>5.6620 | Diminution . . . . 0.0000<br>Further diminution 0.0000<br>" " 0.0000 |
| 8.  | 9.88 per cent iridium.<br>O = 0.5 sq. decim.  | HCl, 20 per cent. T = 80°.                            | I = 10<br>d = 20     | At first . . . .<br>After 70 a.-h.<br>" 140 "            | 5.3367<br>5.3360<br>5.3360           | Diminution . . . . 0.0007<br>Further diminution 0.0000               |
| 9.  | 15.03 per cent iridium.<br>O = 0.5 sq. decim. | HCl, 20 per cent. T = 80°.                            | I = 10<br>d = 20     | At first . . . .<br>After 70 a.-h.<br>" 140 "<br>" 200 " | 5.3700<br>5.3695<br>5.3660<br>5.3658 | Diminution . . . . 0.0005<br>Further diminution 0.0035<br>" " 0.0002 |

The surface O means the total surface of the anodes. I represents the current in ampères; d the density of the current per square decimetre. The computation of the ampère-hours, a.-h., was made, with the exception of Experiment 1, with a copper voltameter by multiplying the constant current I by the length of time measured accurately. T represents the temperature of the electrolyte. The different alloys of platinum and iridium used were made by W. C. Heraeus, and then analysed. In Experiments 1 and 3 we used the same electrode, and, beginning with Experiment 4, a fresh electrode was used every time. The cathodic solution in Experiment 4 was renewed every hour with a fresh concentrated solution of chloride of sodium.

*Experiment 7.*—Before being used the electrode was plunged into concentrated hydrochloric acid for fifteen hours, and there was no diminution of weight.

*Experiment 8.*—The electrode contained 9.88 per cent of commercially pure iridium, the others contained very pure iridium, and no sensible difference could be found in these two kinds of material. The first kind of electrode contains Rh, Ru, Fe, and Cu in much greater quantity than the second; still these are low in both cases, and the experiments do not show any difference; see Mylius and Förster on the preparation of pure platinum, *Berichte*, vol. xxv., p. 665.

*Experiment 9.*—The electrode experienced a considerable diminution in weight, 0.0035 grm., after the second experiment with 70 ampère-hours; this can be explained by the formation of small holes in the electrode, proving its non-homogeneity. Apart from this defect, this electrode behaved like the others.

Experiments 1 and 3 showed that the electrodes used are very resistant to chemical action, as will be seen from the experiments with hydrochloric acid at 20 per cent, and 80°.

Further, it has been observed that with electrodes containing 7.5 to 15 per cent of iridium there was no sensible difference.

To sum up, electrodes of iridio-platinum of 0.007 m.m. in thickness are very resistant to the electrolysis of alkaline chlorides, and their use in ordinary practice can be recommended without hesitation.—*Zeitschrift für Elektrochemie*, vol. viii., p. 147.

**The Use of Organo-magnesian Compounds in Analytical Operations.**—L. A. Tchougaëff. — The organo-magnesian compounds discovered by Grignard, which are of such value in organic synthesis, may be of great use in analytical determinations. We know that the magnesian compounds of the type RMgI decompose water according to the following equation:— $\text{RMgI} + \text{H}_2\text{O} = \text{RH} + \text{MgIOH}$ . Other hydroxylised compounds, such as the alcohols, phenols, and oximes (without mentioning the acids) give an analogous reaction:  $\text{RMgI} + \text{R}_1\text{OH} = \text{RH} + \text{R}_1\text{OMgI}$ . This property may be utilised for a double purpose:—1. As a means for the diagnosis of the hydroxylised compounds. These latter, properly dried, are brought into contact with an excess of the methyl-derivative,  $\text{CH}_3\text{MgI}$ . The reaction can be brought about in a Knop apparatus; the disengagement of methane will indicate the presence of the CH group. 2. As a means for the separation of substances containing hydroxyl from those which do not contain it—for example, for the separation of the alcohols from the hydrocarbides. To the mixture in question we add a slight excess of  $\text{CH}_3\text{MgI}$  in ethereal solution;  $\text{CH}_4$  is given off, and the alcohol passes into the non-volatile form of combination, ROMgI. The carbide is not changed; it can now be separated by distillation under reduced pressure, after evaporating off the ether, and from the residue the alcohol can be re-formed by the action of water on the ROMgI. In this manner the author has already obtained several results of practical utility, and is continuing his researches.—*Journ. Soc. Phys. Chim. R.*, vol. xxxiv., p. 652.

## PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, March 3rd, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MESSRS. W. W. Underhill and J. A. Goodson were formally admitted Fellows of the Society.

The PRESIDENT announced that arrangements had been made for the delivery of a Faraday Lecture this year. The invitation addressed in the first instance to Professor Emil Fischer had been accepted and a date in November last had been selected for the lecture, but to the great regret of the Council the state of Professor Fischer's health had prevented him from fulfilling his engagement. Professor Ostwald, of Leipzig, had, however, been good enough to accept an invitation to give the lecture on April 19th next, and by the courtesy of the authorities of the Royal Institution the discourse would be delivered in the Theatre of that Institution. Arrangements as to the admission of Fellows of the Society would be announced later in the *Proceedings*.

Certificates were read for the first time in favour of Messrs. Robert S. Finlow, Pemberandah, Dalsingh Serai, India; Robert de J. Fleming-Struthers, B.A., Exeter College, Oxford; George Mathieson Horn, Ivylands, Epping; Alfred Mander, Berkswell, Malvern; Gerald Pinchbeck, 96, Albany Street, Regent's Park, N.W.; Walter Tong, Pole Lane, Failsworth, Manchester.

Of the following papers, those marked \* were read:—

\*33. "*Chemical Dynamics of the Alkyl Iodides*." By Miss KATHERINE B. BURKE and FREDERICK GEORGE DONNAN.

The reactions between silver nitrate and certain of the alkyl iodides in absolute alcoholic solutions were shown to be very complicated, and the main conclusions deduced are summarised as follows:—

1. The reactions between silver nitrate and an alkyl iodide in absolute alcohol at 24° 5' and at concentrations varying from N/20 to N/80 can be expressed by a special form of the bimolecular velocity-equation, in which the velocity-coefficient is a function of the initial concentration of the reacting components.

2. It is found that in solutions containing the reagents in equivalent amounts, the velocity-coefficient ( $k$ ) increases as the initial molecular concentration ( $c$ ) increases, the relation between  $k$  and  $c$  being  $k = Kc^{0.53}$ ; where  $K$  is independent of concentration.

3. This variation of  $k$  is chiefly due to the silver nitrate. For solutions containing the same (N/40) initial concentration of ethyl iodide and varying (N/20—N/80) initial concentration ( $c$ ) of silver nitrate, the relation between  $k$  and the latter is approximately  $k = Kc^{\frac{1}{2}}$ . For solutions containing the same initial concentration of silver nitrate and varying initial concentration of ethyl iodide, the value of  $k$  decreases as the latter increases, but the rate of decrease is relatively small as compared with the rate of increase produced by silver nitrate.

4. It has not been found possible to explain the variations of  $k$  indicated in 1, 2, and 3. In particular, the assumption that it is the silver ions which take part in the fundamental reaction which regulates the speed does not appear to offer a simple explanation.

5. Similar anomalies have been observed by Hecht, Conrad, and Brückner in the case of ether-formation from sodium alkyl oxide and alkyl iodide in alcoholic solution, but the explanation of these anomalies proposed by Steger, based on assumptions concerning the ionisation of the sodium alkyl oxide in alcoholic solution, does not appear to be valid if the ordinary law of equilibrium is assumed.

6. The theory of "alkylene" and "alkylidene" dissociation of the alkyl iodides, proposed by Nef, does not give a satisfactory account of the observed results.

7. As stated by Nef, nitric acid is the only acid produced in the reaction. Chiminello's statement that acetic acid is formed appears to be incorrect. The other products of the reaction are ether and alkyl nitrate.

8. If the reactivities of the alkyl iodides are measured by the velocity-coefficients of the reaction with silver nitrate in absolute alcohol (N/40 equivalent solutions at 24° 5'), the order of reactivity (beginning with the greatest) is isopropyl, ethyl, *n*-propyl, methyl, *n*-butyl, isoamyl, isobutyl.

9. This order of reactivities corresponds with the rate of production of free iodine in the alcoholic solutions when exposed to air and light, with the single exception of methyl iodide, the solution of which becomes discoloured at a rate between those of solutions of isopropyl and ethyl iodides.

10. The relative reactivities referred to in 8 do not agree in many respects with those observed in other reactions which have been studied kinetically, such as the reactions with ethyl sodioacetate, triethylamine, sodium alkyl-oxide. Compared with these results, isopropyl iodide reacts with abnormally great, and methyl iodide with abnormally small velocity. It does not appear possible, therefore, to ascribe the reactivity of the alkyl haloids to any uniform cause (such, for example, as a dissociation, whether "alkylidene," "alkylene," or electrolytic).

11. So far as the final products and not the kinetics of the reaction are concerned, the production of ether and nitric acid can be explained by ionic reactions just as well as by Nef's hypothesis.

\*34. "*Separation of  $\beta$ -Crotonic Acid from  $\alpha$ -Crotonic Acid*." By ROBERT SELBY MORRELL and ALBERT ERNEST BELLARS.

The separation of  $\beta$ -crotonic acid from  $\alpha$ -crotonic acid was formerly effected by means of the different solubilities of the sodium salts in alcohol (Michael and Schulthess, *Journ. Pr. Chem.*, 1892, [2], xlvii., 245), the  $\beta$ -crotonate being more soluble in absolute alcohol than its  $\alpha$ -isomeride. The  $\beta$ -crotonic acid thus obtained was considered by J. Wislicenus to be not entirely free from the  $\alpha$ -acid (*Chem. Centr.*, 1897, ii., 259).

The salts of  $\beta$ -crotonic acid with brucine and quinine have been found to be less soluble than the corresponding derivatives of  $\alpha$ -crotonic acid. The brucine salts are unfortunately very soluble in most solvents, but the quinine salts are very sparingly soluble in water, and it is easy to separate quinine  $\beta$ -crotonate (m. p. 157°) from quinine  $\alpha$ -crotonate (m. p. 134°) by fractional crystallisation; the solubility of the  $\beta$ -crotonate in water at 17° is 1:0, whilst that of the  $\alpha$ -crotonate is 2:4 at the same temperature, and two crystallisations are sufficient to complete the separation.

The barium  $\beta$ -crotonate,  $\text{Ba}(\text{C}_4\text{H}_5\text{O}_2)_2 \cdot \text{H}_2\text{O}$ , obtained from the quinine salt, when suspended in pure ether and decomposed by dilute sulphuric acid, yielded the pure  $\beta$ -crotonic acid (m. p. 15°, sp. gr. 1.0342 at 12.5°). Its physical and chemical properties agree with those described by Michael and by J. Wislicenus (*loc. cit.*). The molecular weight in glacial acetic acid is 85.6, and the molecular refraction is 37.2.

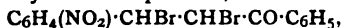
The advantage of the following method of separation lies in the fact that fractional distillation of the two acids is avoided, and it is easier to obtain the less soluble quinine  $\beta$ -crotonate in a pure state than to remove sodium  $\alpha$ -crotonate completely from the more soluble sodium  $\beta$ -crotonate.

\*35. "*Contributions to the Knowledge of the  $\beta$ -Diketones*." By SIEGFRIED RUHEMANN and EDWIN ROY WATSON.

The authors have repeated Wislicenus's experiments on the action of alcoholic potash in benzylideneacetophenone dibromide, and find that the compound produced has the empirical formula  $\text{C}_{17}\text{H}_{16}\text{O}_2$ , and not  $\text{C}_{15}\text{H}_{12}\text{O}_2$ , as stated by Wislicenus (*Annalen*, 1899, cccviii., 219), and that it is the ethyl ether of dibenzoylmethane,  $\text{C}_6\text{H}_5\cdot\text{C}(\text{O}\cdot\text{C}_2\text{H}_5)\cdot\text{CH}\cdot\text{CO}\cdot\text{C}_6\text{H}_5$ . Under the influence of

hydrochloric acid, the substance decomposes into ethyl chloride, acetophenone, and benzoic acid.

Alcoholic potash behaves similarly towards the dibromide of *p*-nitrobenzylideneacetophenone,—



yielding the ethyl ether of *p*-nitrobenzoylmethane,  $\text{C}_6\text{H}_4(\text{NO}_2) \cdot \text{C}(\text{O} \cdot \text{C}_2\text{H}_5) : \text{CH} \cdot \text{CO} \cdot \text{C}_6\text{H}_5$  (m. p. 89–90°).

The authors have also examined the action of ammonia and organic bases on the olefinic diketones. Thus benzylideneacetylacetone reacts with aniline to form the compound  $\text{C}_6\text{H}_5 \cdot \text{CH}(\text{NH} \cdot \text{C}_6\text{H}_5) \cdot \text{CH}(\text{CO} \cdot \text{CH}_3)_2$  (m. p. 113°). This, on heating, decomposes into benzylideneaniline and acetylacetone. Phenylhydrazine reacts with benzylideneacetylacetone and also with benzylidenebenzoylacetone with evolution of heat. The additive compounds could not be obtained, since, in each case, they are decomposed at once with the formation of benzaldehyde phenylhydrazone.

With benzylideneacetylacetone, alcoholic ammonia forms the compound  $\text{C}_{19}\text{H}_{20}\text{ON}_2$  (m. p. 147°), which is to be regarded as acetyldiphenylmethyltetrahydropyrimidine,  $\text{C}_6\text{H}_5 \cdot \text{CH} \left\langle \begin{array}{c} \text{C}(\text{CO} \cdot \text{CH}_3) : \text{C}(\text{CH}_3) \\ \text{NH} \text{---} \text{CH}(\text{C}_6\text{H}_5) \end{array} \right\rangle \text{NH}_3$ .

The diketones react with semicarbazide with the elimination of 1 molecule of water and the formation of  $\text{C}_{13}\text{H}_{15}\text{O}_2\text{N}_3$  (m. p. 210° with decomposition) and  $\text{C}_{18}\text{H}_{17}\text{O}_2\text{N}_3$  (decomposing at 230°). These compounds may be regarded as semicarbazones, but taking into consideration the behaviour of other bases towards the diketones, the alternative view of representing them as cyclic compounds cannot be disregarded.

The additive compound obtained from benzamidine and benzylidenebenzoylacetone (*Trans.*, 1903, lxxxiii., 1372) may also be regarded as being formed by the addition of the base to the ethylenic linking of the olefinic ketone.

\*36. "The Purification of Water by continuous Fractional Distillation." By WILLIAM ROBERT BOUSFIELD.

An apparatus was described in which water is distilled continuously from a copper boiler, freed from spray by means of a system of baffle plates, and condensed on surfaces kept at different temperatures by an adjustable supply of cold water. The hottest surfaces yield distilled water of ordinary quality, whilst the coldest surfaces give water of conductivity 1 to 2 reciprocal megohms per c.c., which is sufficiently pure for the majority of conductivity measurements, and contains only a small proportion of ammonia.

\*37. "Freezing-point Curves of Dynamic Isomerides. Ammonium Thiocyanate and Thiocarbamide." By ALEXANDER FINDLAY.

The author has studied the equilibrium relations of the dynamic isomerides, ammonium thiocyanate, and thiocarbamide from the point of view of the phase rule, attention being directed chiefly to the determination of the freezing-points of mixtures of these substances with the view of ascertaining what compounds, if any, are formed at temperatures in the neighbourhood of the freezing-points, and the conditions of their stability (compare Emerson Reynolds and Werner, *Trans.*, 1903, lxxxiii., 1). The freezing-point curve obtained is of the simplest form, consisting of two branches meeting at a eutectic-point which lies about 104.3°. The melting-points of ammonium thiocyanate and of thiocarbamide can be determined only approximately on account of the occurrence of isomeric transformation. The melting-point of ammonium thiocyanate may be taken as being about 149° and that of thiocarbamide as not lower than 175–177°, these being determined by comparison with camphor. The natural freezing-point is 114–115°, the stable form being ammonium thiocyanate. From the simple form of the freezing-point curve obtained, it follows that no compound is formed which is stable at the temperatures indicated on this curve. This conclusion is supported by the form of the cooling curve and by the analysis of the solid phase.

From the fact that the equilibrium-point in the liquid phase is independent of the temperature, it follows that the

isomeric transformation is not accompanied by any heat effect.

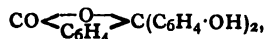
38. "The Constitution of Phenolphthalein." By ARTHUR GEORGE GREEN and ARTHUR GEORGE PERKIN.

The authors have found that a solution of phenolphthalein, decolorised by an excess of caustic alkali, can be entirely neutralised without any return of the colour taking place, by careful titration at a low temperature with dilute acetic acid. If, however, the colourless neutral solution is boiled, the red colour returns in its full intensity, whilst at the same time the solution becomes alkaline. If the solution is acidified and either left for some time or heated, a precipitation of free phenolphthalein occurs, which also dissolves in aqueous alkalis to a red solution. The point at which neutrality occurs in the titration with acetic acid corresponds with the presence in the colourless solution of the carbinolcarboxylic acid salt,  $\text{C}_6\text{H}_4(\text{CO}_2\text{M}) \cdot \text{C}(\text{OH})(\text{C}_6\text{H}_4 \cdot \text{OH})_2$ .

These observations do not agree with the electrolytic dissociation hypothesis, but are simply explained by the quinonoid theory if the colour changes are attributed to a variation of type from a quinonoid to a benzenoid form and *vice versa*, due to hydration and dehydration.

Thus, the first action of an alkali on the free phenolphthalein (lactone) would probably be the replacement of the phenolic hydrogen by the alkali metal. This salt being unstable may be supposed to undergo immediate transformation into the coloured quinonoid salt,  $\text{C}_6\text{H}_4(\text{CO}_2\text{K}) \cdot \text{C}(\text{C}_6\text{H}_4 \cdot \text{OH}) : \text{C}_6\text{H}_4 \cdot \text{O}$ , by direct transference of the metal from the phenolic to the carboxylic group.

With excess of caustic alkali, this coloured salt is converted, by assumption of KOH, into the colourless carbinol salt,  $\text{C}_6\text{H}_4(\text{CO}_2\text{K}) \cdot \text{C}(\text{OH})(\text{C}_6\text{H}_4 \cdot \text{OH}) \cdot \text{C}_6\text{H}_4 \cdot \text{OK}$ , and this product, when neutralised with acetic acid, gives  $\text{C}_6\text{H}_4(\text{CO}_2\text{K}) \cdot \text{C}(\text{C}_6\text{H}_4 \cdot \text{OH})_2 \cdot \text{OH}$ , which is also colourless. Caustic potash is set free on boiling the latter salt, and the lactone,—



which is produced is simultaneously converted into the coloured quinonoid salt by the liberated alkali.

A similar explanation is offered in the case of quinolphthalein, the behaviour of which is found to be exactly similar to that of phenolphthalein.

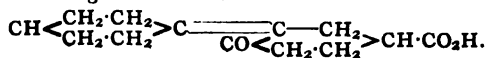
39. "δ-Ketohexahydrobenzoic Acid." By WILLIAM HENRY PERKIN, jun.

Ethyl γ-cyanopentane-α,γ-tricarboxylate,—



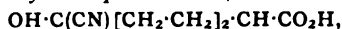
is readily prepared by the interaction of ethyl β-iodopropionate with the sodium compound of ethyl cyanoacetate; it boils at 228° under 20 m.m. pressure, and, on hydrolysis with hydrochloric acid, yields pentane-α,γ-tricarboxylic acid,  $\text{CO}_2\text{H} \cdot \text{CH}(\text{CH}_2 \cdot \text{CH}_2 \cdot \text{CO}_2\text{H})_2$  (compare Emery, *Ber.*, 1891, xxiv., 384; Bottomley and Perkin, *Trans.*, 1900, lxxvii., 299). On digesting this acid with acetic anhydride and distilling the product under diminished pressure, water and carbon dioxide are eliminated and δ-ketohexahydrobenzoic acid (cyclohexanone-4-carboxylic acid) is produced, which crystallises with one molecule of water, melts at 68°, and distils at 210°/30 m.m. It was further characterised by means of its oxime, semicarbazone, and methyl and ethyl esters.

The ketonic acid forms an oily phenylhydrazone, which, on heating with hydrochloric acid, yields tetrahydrocarbazole-*p*-carboxylic acid (m. p. 195°); compare Baeyer and Tutein, *Ber.*, 1899, xxii., 2184). δ-Ketohexahydrobenzoic acid, when digested with methyl alcohol and sulphuric acid, yields ethyl carboxyhexamethenyl-δ-ketohexahydrobenzoate (b. p. 255°/20 m.m.). This ester, on hydrolysis, yields the acid, which melts at 170°, and has the following constitution:—



trans-Hydroxyhexahydrobenzoic acid, produced by reducing  $\delta$ -ketohexahydrobenzoic acid, melts at  $121^{\circ}$ , and, when heated with hydrobromic acid, is converted into trans- $\delta$ -bromohexahydrobenzoic acid, (bromocyclohexane-4-carboxylic acid) (m. p.  $167^{\circ}$ ), and this, when digested with sodium carbonate, loses hydrogen bromide, yielding  $\Delta^8$ -tetrahydrobenzoic acid, which melts at about  $13^{\circ}$  and distils at  $237^{\circ}/748$  m.m.; it combines with hydrobromic acid to form  $\gamma$ -bromohexahydrobenzoic acid, (m. p.  $122^{\circ}$ ), and also absorbs bromine at the ordinary temperature, yielding  $\gamma\delta$ -dibromohexahydrobenzoic acid (m. p.  $86^{\circ}$ ).

$\delta$ -Ketohexahydrobenzoic acid combines with hydrogen cyanide, and from the product, the nitrile of trans- $\alpha$ -hydroxyhexahydroterephthalic acid,—



was isolated in the form of pale yellow crystals melting at about  $140^{\circ}$ . This nitrile yields, on hydrolysis, trans- $\alpha$ -hydroxyhexahydroterephthalic acid (m. p.  $228$ — $230^{\circ}$ ). If the crude product of the action of hydrogen cyanide on ketohexahydrobenzoic acid is hydrolysed, the principal product obtained is  $\alpha$ -hydroxyhexahydroterephthalic acid, (m. p.  $168$ — $170^{\circ}$ ).

Both *cis*- and *trans*-hydroxy-acids are decomposed on distillation, with loss of water and formation of  $\Delta^1$ -tetrahydroterephthalic acid (Baeyer, *Annalen*, 1888, ccxlv., 160).

The investigation of  $\delta$ -ketohexahydrobenzoic acid is being continued.

(To be continued).

## NOTICES OF BOOKS

*The Grant and Validity of British Patents for Inventions.*

By JAMES ROBERTS, M.A., LL.B., &c. With many Diagrams. London: John Murray. 1903. Pp. 647.

It has been said that the granting of a patent gives the inventor no privilege beyond that of defending its validity in a court of law. This is perhaps rather overstating the case, still it must be admitted that the English Patent Law is in a very unsatisfactory condition. We find it stated in this volume that no fewer than 42 per cent, or about 5780 patents granted per annum in England, are invalid on the ground of having been already patented in this country. An examination of the results of litigation shows that no less than 51 per cent of those patents which are commercially worth infringing are invalid; thus about £23,120 per annum is paid for patents which give no legal protection to the patentees.

A new procedure is about to be introduced which will prevent the granting of a large proportion of bad patents. Under this new procedure, questions of alleged anticipation will arise, and these questions will have to be considered by the inventor before, instead of as heretofore after the grant is made.

This work, which is divided into three principal parts, deals with the subject up to the grant of the patent and the amendment of specifications, and is of necessity a law book.

Part I. deals with the general principles and rules affecting the grant and validity of patents, and contains nine chapters.

Part II. consists of abstracts of leading and illustrative cases with notes; this part covers nearly 300 pages, and illustrates the application of the principles in Part I.

Part III. deals with the statutes and rules, in so far as they bear on the grant of patents.

It is stated by the author that the book has been written for inventors, but we can hardly believe that many inventors would take the trouble to consult it; moreover, if they did they would probably not be able to follow it. As we remarked above, it is essentially a law book, and as such will no doubt be of great service to patent agents and practising lawyers, who make a speciality of this particular branch of their profession.

*Drugs, their Production, Preparation, and Properties.* By H. WIPPELL GADD, F.C.S. &c. London: Baillière, Tindall, and Cox. 1904. Pp. 180.

ALTHOUGH this book has been written primarily for students, it will also be found useful by others who occasionally need information, or wish to refresh their memories concerning medicinal products. It embraces all the drugs included in the Pharmacopœia, and they have been arranged in classes, like being placed with like.

The book is divided into three sections, dealing respectively with (1) vegetable, (2) animal, and (3) chemical products, and is provided with a good index and table of contents.

*Dispensing made Easy*; with numerous Formulæ, and Practical Hints to secure Simplicity, Rapidity, and Economy. By WM. G. SUTHERLAND, M.B. (Aberd.) Bristol: John Wright and Co. 1904. Pp. 102.

IN most aids to dispensing a good deal of space is taken up with such subjects as pill-rolling, pill-coating, the making of emulsions, &c., subjects in which the dispensing medical practitioner takes little or no interest. The present volume has been compiled with the object of rendering dispensing as easy as possible, especially to those who have a large club, colliery, or works practice. Economy as well as rapidity is necessary in a practice of the kind just mentioned, and many a useful hint in this direction will be found in these pages.

*The Cyanide Process of Gold Extraction.* A Text-book for the Use of Mining Students, Metallurgists, and Cyanide Operators. By JAMES PARK, Professor of Mining and Director of Otago University School of Mines. Third English Edition, Revised and Enlarged. With Frontispiece, Plates, and Illustrations. London: Charles Griffin and Co., Ltd. 1904. Pp. 193.

THE rapid succession of new editions of this book bear ample evidence as to its value; the first English edition was only published in 1900, and we have already to do with the third.

In this edition much new material has been added, for the most part relating to the lead-smelting of gold slimes, the treatment of sulpho-telluride ores, and filter-press practice.

The value of filter-pressing of course varies with the different gold-fields. In Western Australia its adoption was mainly determined by a combination of local conditions; the tendency of the ores to form slimes, the scarcity of fresh water, and the saline character of the only water available for milling; these slimes were of high value. On the other hand, in South Africa the mill-slimes are only a secondary consideration, and are of low grade.

The adoption of lead-smelting for gold-slimes is a notable advance in cyanide practice; it is best described as scorification on a large scale, for the reason that the zinc-slimes are melted and the gold recovered in lead bullion; this is then cupelled, or rather, refined. It is claimed that lead-smelting recovers a larger amount of gold than the sulphuric acid method, and in six trials the lead process gave 10.5 per cent higher recovery than the acid treatment.

With regard to the cyanide process as a whole, it has been more largely adopted in the Witwatersrand goldfields than anywhere else. The ore is principally a pyritic silicious quartz conglomerate, and the gold is disseminated throughout the matrix or in the iron pyrites; hence the cyanide process is the most suitable one for its recovery.

In some goldfields, notably the Kalgoorlie, sulpho-telluride ores are met with in large quantities; this requires a modification of the cyanide process, such as the use of cyanogen bromide, &c., the merits of which have been already discussed in these columns.

We can confidently recommend this book as a thoroughly practical work, and congratulate the author on its continued success.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 7, February 15, 1904.

**Action of Reduced Nickel on the Halogen Derivatives of the Fatty Series in presence of Hydrogen.**—Paul. Sabatier and Alph. Mailhe.—A series of experiments on the action of reduced nickel in presence of hydrogen on halogen derivatives of the fatty series show that the halogen is in every case more or less easily eliminated in the form of hydric acid, but it is never replaced by the hydrogen, as is the case in the aromatic series. The result is either the formation of an incomplete compound or the complete destruction of the molecule.

**Influence of Complex Ions in Electrolysis by Alternating Currents.**—André Brochet and Joseph Petit.—In a previous research the authors proved that the formation of a complex ion is not the true cause of the solution of copper in potassium cyanide under the influence of an alternating current. They now consider this solution as a case analogous to that of the solution of pure zinc in sulphuric acid—an action which becomes more rapid under the influence of an alternating current. Other complex ions which can either be formed or destroyed under the influence of a continuous current are also formed by means of an alternating current—as in the case of copper, nickel, and cobalt dissolving in ammoniacal salts (carbonate, sulphate, or chloride). However, as the spontaneous action is zero, the reaction is less active than in the case of the cyanide, and the yields proportionately less.

**$\gamma$ -Chloroacetylacetic Ether.**—M. Lespieau.—The author prepares  $\gamma$ -chloroacetylacetic ether,  $\text{CH}_2\text{Cl}.\text{CO}.\text{CH}_2.\text{CO}_2\text{C}_2\text{H}_5$ . He finds it to be a colourless liquid of density 1.23, boiling at  $105^\circ$  at pressure 11 m.m. It distils at about  $205^\circ$  under atmospheric pressure, but considerable decomposition then takes place. It colours dilute solutions of iron persalts a dark reddish violet. Its copper salt,  $(\text{CH}_2\text{Cl}.\text{CO}.\text{CH}.\text{CO}_2\text{C}_2\text{H}_5)_2\text{Cu}$ , crystallised from benzene, is anhydrous, of bright green colour, and decomposable about  $160^\circ$ .

**Dichloromethene-dioxypropylbenzene and Propylpyrocatechin Carbonate.**—R. Delange.—Dichloromethene-dioxypropylbenzene is an extremely active body which reacts immediately on water, alcohols, phenols, acids, acid anhydrides, ammonia, amines, &c. When carbon oxychloride acts on disodium propylpyrocatechin, the diphenolic carbonate is directly formed—a reaction which absolutely establishes the composition of the latter to be  $\text{C}_3\text{H}_7-\text{C}_6\text{H}_4-\text{O} > \text{CO}$ .

**Glyoxylic Ureides. Allantoin and Allantoic Acid.**—L. J. Simon.—Allantoin is soluble in aqueous potash. On immediate acidification it is reprecipitated without loss or alteration. If, however, the potash solution is left for some days, the solution contains a new potassium salt. Müller isolated this, analysed it, and called it potassium allantolate. Ponomareff isolated allantoinic acid itself by utilising dilute sulphuric acid for the neutralisation. Finally, Grimaux effected the synthesis of allantoin by the action of glyoxylic acid on urea at  $100^\circ$ , without being able to isolate allantoinic acid, which he considered as an intermediary in this synthesis. The author now repeats all these experiments, and prepares glyoxylic ureids—allantoin and allantoinic acid.

**Composition of Potato Starch.**—A. Fernbach.—Already noticed.

## MISCELLANEOUS.

**Royal Institution.**—The following are the lecture arrangements at the Royal Institution, after Easter:—Prof. L. C. Miall, three lectures on "The Transformation of Animals"; Mr. L. Fletcher, three lectures on "Meteorites"; Mr. H. F. Newall, two lectures on the "Solar Corona"; Prof. Dewar, three lectures on "Dissociation"; Mr. H. G. Wells, two lectures on "Literature and the State"; Mr. Cyril Davenport, three lectures on (1) "Mezzotints," (2) "Cameos," (3) "Jewellery"; Mr. Donald Francis Tovey, three lectures on the "Sonata" (with musical illustrations); and Sir W. Martin Conway, two lectures on "Spitzbergen in the Seventeenth Century." The Friday Evening Meetings will be resumed on April 15, when Monsignor the Count v. de Vaya and Luskod will deliver a discourse on "Korea and the Koreans." Succeding discourses will probably be given by Lieut.-Col. David Bruce, the Dean of Westminster, Dr. P. Chalmers Mitchell, Mr. M. H. Spielmann, Prof. E. Rutherford, H.S.H. the Prince of Monaco, Prof. S. Arrhenius, and other gentlemen.

**Aqueous Solutions of Colloidal Selenium.**—A. Gutbier.—Solutions of colloidal selenium, obtained for the first time by Schulze (*Journ. Prakt. Chem.*, [2], vol. xxxii., p. 390), are obtained by treating solutions of selenous acid with hydrate of hydrazine (1 part in 2000), hydrochlorate of hydroxylamine, or hypophosphorous acid. The solutions are red by transmitted light and a fluorescent blue by reflected light. They can be concentrated and filtered without changing; when submitted to electrolysis they deposit red selenium. By prolonged concentration of the solutions *in vacuo* over sulphuric acid, soluble colloidal selenium (hydrosal) is deposited, but more often the major portion is transformed into the insoluble colloidal selenium (hydrogel).—*Zeit. Anorg. Chem.*, vol. xxxii., p. 106.

**Aqueous Solution of Colloidal Gold.**—A. Gutbier.—For the preparation of such a solution, we dissolve 1 grm. of  $\text{AuCl}_3$  in 1 litre of distilled water, and add carbonate of soda until neutral. The solution is then treated in the cold, and slowly, with a few drops of a very dilute solution of hydrate of hydrazine (the commercial solution at 50 per cent diluted with 2000 volumes of water). An excess of the hydrazine solution must be avoided. The colloidal solution is deep blue in colour both by reflected and by transmitted light. Its colour is similar to that of indigo. If by reflected light the liquor has a golden appearance it is because too much hydrazine has been added, and in such a case the precipitation of the gold takes place at once. The solutions are easily dialysed, and are very stable. Electrolysis causes the precipitation of the metal.—*Zeit. Anorg. Chem.*, vol. xxxi., p. 448.

**Gravimetric Estimation of Tellurium by means of Hypophosphorous Acid.**—A. Gutbier.—Even with very dilute solutions of telluric acid, hypophosphorous acid gives a brownish blue colouration, and thus enables us to detect tellurium qualitatively in solutions which do not contain the heavy metals, and which are free from free sulphuric or nitric acids; on the other hand, the presence of hydrochloric acid facilitates the reaction. For the quantitative estimation of the tellurium, the solution is treated with its own volume of hypophosphorous acid (a 50 per cent solution). Heat until the liquid, which is at first brown or blue in colour, becomes completely limpid. The precipitated tellurium is dried at  $105^\circ$  until the weight is constant. Instead of 55.57 and 79.95 of tellurium, the author obtained figures comprised between 55.20–55.82 and 79.84–80.05.—*Zeit. Anorg. Chem.*, vol. xxxii., p. 295.

**Iodimetric Estimation of Bismuth in the Form of Chromate.**—E. Rupp and O. Schaumann.—The method described by Mohr in his Treatise on Analysis (seventh edition, p. 241, 1896) for the volumetric estimation of



bismuth does not give satisfactory results. This method, which consists of precipitating the bismuth by means of  $\text{Cr}_2\text{O}_7\text{K}_2$ , collecting the chromate of bismuth and titrating it with ammoniacal sulphate of iron, can be made perfectly exact by operating as follows:—The solution of bismuth containing the smallest possible quantity of free acid is poured into a known volume of a titrated solution of  $\text{CrO}_4\text{K}_2$  (at about 5 per cent). The solution is then diluted, shaken energetically, and after ten minutes it is filtered. After making certain that the whole of the bismuth is precipitated, the excess of  $\text{CrO}_3$  is estimated on a portion of the filtrate, by means of  $\text{KI} + \text{SO}_4\text{H}_2$ ; the iodine set free is then titrated by means of  $\text{S}_2\text{O}_3\text{Na}_2$ .—*Zeit. Anorg. Chem.*, vol. xxxiii., p. 359.

### MEETINGS FOR THE WEEK.

MONDAY, 21st.—Society of Arts, 8. (Cantor Lectures). "Recent Advances in Electro-chemistry," by Bertram Blount, F.I.C.

TUESDAY, 22nd.—Royal Institution, 5. "The Doctrine of Heaven and Hell in Ancient Egypt, and the Books of the Underworld," by E. A. Wallis Budge, M.A., &c. Society of Arts, 4.30. "Cotton Growing in the British Empire," by Alfred Emmott, M.P.

WEDNESDAY, 23rd.—Society of Arts, 8. "The Rural Housing Question," by T. Brice Phillips.

THURSDAY, 24th.—Royal Institution, 5. "Shakespeare as Contemporaries knew him," by Sidney Lee, Litt.D.

FRIDAY, 25th.—Royal Institution, 9. "Liquid Hydrogen Calorimetry," by Prof. Dewar, F.R.S.

Physical, 5. "Note on the Measurement of Small Inductances and Capacities and on a Standard of Small Inductance" and "A Hot-wire Ammeter for Measuring very small Alternating Currents," by Prof. J. A. Fleming, F.R.S. "The Energy of Secondary Röntgen Radiation," by C. G. Barkla.

SATURDAY, 26th.—Royal Institution, 3. "The Life and Work of Stokes," by Right Hon. Lord Rayleigh, F.R.S.

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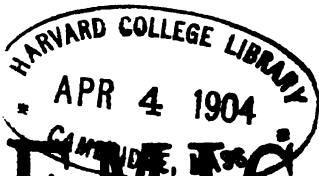
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### CONTENTS.

| ARTICLES:—                                                                                                                        | PAGE |
|-----------------------------------------------------------------------------------------------------------------------------------|------|
| Note on the Formation of Solids at Low Temperatures, particularly with regard to Solid Hydrogen, by Morris W. Travers, D.Sc. .... | 145  |
| London Water Supply, by Sir William Crookes, F.R.S., and Prof Dewar, F.R.S. ....                                                  | 146  |
| The Precipitation of Magnesium Oxalate with Calcium Oxalate, by Nicholas Knight. ....                                             | 146  |
| Contributions to the Chemistry of the Rare Earths, by Chas Baskerville and J. W. Turrettine. ....                                 | 147  |
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S. ....                                                  | 150  |
| PROCEEDINGS OF SOCIETIES—                                                                                                         |      |
| CHEMICAL SOCIETY. ....                                                                                                            | 152  |
| PHYSICAL SOCIETY. ....                                                                                                            | 153  |
| NOTICES OF BOOKS. ....                                                                                                            | 154  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES. ....                                                                                       | 155  |
| MISCELLANEOUS. ....                                                                                                               | 156  |

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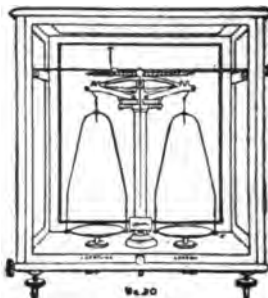
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2313.

## NOTE ON THE FORMATION OF SOLIDS AT LOW TEMPERATURES, PARTICULARLY WITH REGARD TO SOLID HYDROGEN.\*

By MORRIS W. TRAVERS, D.Sc.,  
Professor of Chemistry at University College, Bristol.

IN the year 1902 Dr. Jaquerod and I carried out some experiments on liquid and solid hydrogen with a view to determining its vapour pressure on the scales of the constant-volume helium and hydrogen thermometers. We found that hydrogen remained liquid down to  $14.2^{\circ}$  (He scale), the lowest temperature to which we could reduce a large mass of the liquid by means of the pump at our disposal. When, however, a small quantity of liquid hydrogen, cooled to  $14.2^{\circ}$  in a glass tube immersed in the liquid contained in the large vacuum vessel, was allowed to evaporate under reduced pressure, it solidified when the pressure fell to 49 or 50 m.m. of mercury. This pressure corresponds to a temperature of  $14.1^{\circ}$  on the helium scale. The presence of the solid was determined by mechanical means, and it was not possible to observe its appearance (*Phil. Trans.*, A, vol. cc., p. 170).

Dewar gives the melting-point of hydrogen at about  $15^{\circ}$  absolute, and the melting-pressure at 55 m.m. of mercury. He describes its appearance as that of "frozen foam," or as "clear transparent ice" (British Association, Presidential Address, 1902; see also paper on "Solid Hydrogen," *Brit. Assoc. Report*, 1899, reprinted in *Nature*; also *Roy. Inst. Proc.*, 1900).

It appeared to me worth while to carry out a few experiments to try to determine whether solid hydrogen formed definite crystal, or indeed whether the glassy substance was a true solid or merely a highly viscous fluid. My meaning will become clearer if I give an instance in which both such changes occur.

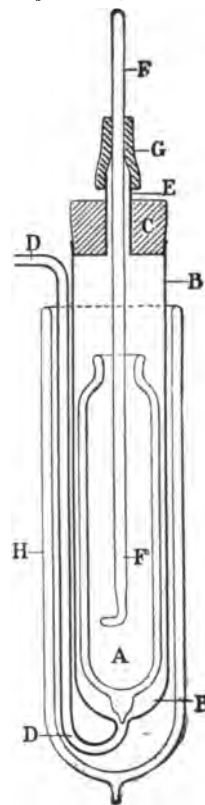
If an organic liquid, such as ethyl aceto-acetate, is cooled slowly to the temperature of liquid air it is converted into crystalline solid, the formation of the crystals commencing when the liquid is cooled to about  $-150^{\circ}$  C., usually at several points on the side of the vessel, and spreading rapidly throughout the mass. If, on the other hand, the liquid is cooled very rapidly, a hard glassy substance is formed, and though crystals may begin to appear, they will only do so locally, as the velocity of crystallisation decreases rapidly as the viscosity of the liquid increases. The glassy substance is really a liquid of high viscosity; it is formed with perfect continuity from the normal liquid state, and should differ from the solid (crystalline) form in its physical properties. Such a substance might, for convenience, be called a pseudo-solid.

In investigating solid hydrogen the apparatus shown in the accompanying figure was employed. The liquid hydrogen was introduced into a small clear glass vacuum-vessel 15 c.m. long and 4 c.m. in internal diameter. This vessel was placed inside a glass tube, B B, which communicated with an exhaust pump through a tube, D D, sealed to it, and was closed by a rubber stopper, C. A short glass tube, E, 6 m.m. in diameter, passed through the stopper, and through it passed the stirring rod, F F. To allow of free rotating motion to the stirrer, and to make the apparatus gas-tight, a short piece of rubber tube, G, was passed over the end of the tube, E, and was wired to F. The lower part of the apparatus was contained within the vacuum vessel, H, which contained a small quantity of liquid air.

When the liquid hydrogen was made to boil *in vacuo*, its temperature fell, but the liquid did not appear to become more viscous. At length films of a colourless glassy sub-

stance formed at the surface, and broke away as the bubbles rose. After a short time the vessel became filled with these flakes, and while in this condition stirring, by giving the top of the rod F a rotatory motion, did not appear to indicate that the portion which remained liquid had undergone any considerable increase in viscosity. After a time the mass contained so much solid that it became pasty, and finally the whole of it appeared fairly homogeneous.

The solid evaporated fairly rapidly, so that after about ten minutes only a hollow cylinder of it, about 3 c.m. long and 2.5 c.m. in diameter, remained. This had the appearance of a film of ice which had partly thawed, consisting of clear granules connected by thinner and less transparent portions of solid. No crystals were observed on either of the three occasions on which the experiments were carried out. An attempt was made to examine the solid in the field of a polariscope, but it was unsuccessful.



Though there is no direct evidence of the formation of crystalline hydrogen, my experiments led me to the belief that solid hydrogen is a crystalline substance and not a pseudo-solid. The sharpness with which the solid hydrogen is formed, and the constancy of the apparent melting pressure, are distinct evidence in favour of this conclusion, though it must be allowed that the rate of change in viscosity, when the temperatures are measured on the Centigrade scale, will probably appear to be more rapid at low temperatures than at high temperatures.

The whole question of the formation of solids at very low temperatures is of great interest both from a physical and from a biological standpoint. It is quite possible that if living organisms were cooled only to temperatures at which physical changes such as crystallisation take place with measurable velocity, the process would be fatal, whereas if they once were cooled to the temperature of liquid air, no such change could take place within finite time, and the organism would survive. (Experimental results are given by Macfadyen, *Proc. Roy. Soc.*, 1900, vol.

\* A Paper read before the Royal Society, February 18th, 1904.

lxvi., pp. 180, 339, 488; Swithinbank, *Proc. Roy. Soc.*, 1901, vol. lxxviii., p. 502).

These experiments were made in connection with some investigations which were being carried out at University College, London, with the assistance of a grant from the Royal Society. As I am at the moment unable to continue the work, I have decided to publish this note.

### LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING FEBRUARY 29TH, 1904.

By SIR WILLIAM CROOKES, F.R.S.,  
and  
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,  
*Water Examiner, Metropolis Water Act, 1871.*

London, March 10th, 1904.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 203 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Feb. 1st to Feb. 29th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 203 samples examined by us during the month, all were clear, bright, and well filtered.

There has been a further excess of rain at Oxford during the past month; the actual rainfall measured was 2·98 inches. The average fall for February is 1·80 inches; thus we have an excess of 1·18 inches, which, if added to the previous excess for January, makes a total excess of 2·20 inches, or 56·7 per cent above the thirty-five years' average.

Our bacteriological examinations of 358 samples taken during last month have given the results recorded in the following table. Besides these samples we have examined 399 others from special wells, standpipes, &c., making 757 samples in all:—

|                                                                                                                 | Microbes<br>per c.c. |
|-----------------------------------------------------------------------------------------------------------------|----------------------|
| New River, unfiltered (mean of 25 samples) ..                                                                   | 848                  |
| New River, filtered (mean of 68 samples) ..                                                                     | 33                   |
| Thames, unfiltered (mean of 25 samples) ..                                                                      | 19530                |
| Thames-derived water from the clear-water<br>wells of eight Thames-derived supplies (mean<br>of 192 samples) .. | 39                   |
| Ditto ditto .. .. . highest                                                                                     | 434                  |
| Ditto ditto .. .. . lowest                                                                                      | 0                    |
| River Lea, unfiltered (mean of 25 samples) ..                                                                   | 347                  |
| River Lea, from the East London Water Com-<br>pany's clear-water wells (mean of 25 samples)                     | 27                   |

Of the 285 daily samples taken from the general filter wells of the Metropolitan Water Companies, seven samples, or 2·4 per cent, were sterile. Twenty-seven samples, or 9·4 per cent, contained more than 100 microbes, and of these, ten samples contained more than 150 microbes per c.c. The twenty-seven excess samples contained an average of 162 microbes per c.c. In January ten excess samples contained an average of 119 microbes per c.c.

The considerable increase in the number of microbes in the filtered water during the past month has been caused undoubtedly by the excessive rainfall and the flooded con-

dition of the Thames Valley. In January the unfiltered Thames water contained an average of 8797 microbes; in February this figure has risen to 19,530, while the numbers in the filtered samples have increased from 21 to 39. Considering the unfavourable condition of the Thames and Lea, the distribution of a filtered water containing no more than an average of 40 microbes per c.c. may be considered, under present circumstances, satisfactory.

We are, Sir,

Your obedient Servants,  
WILLIAM CROOKES.  
JAMES DEWAR.

### THE PRECIPITATION OF MAGNESIUM OXALATE WITH CALCIUM OXALATE.

By NICHOLAS KNIGHT.

THE purpose of this work was to determine the amount of magnesium oxalate that will precipitate with calcium oxalate in connection with the analysis of dolomite rock by different students. These rocks abound in Iowa and in many other portions of the United States. The specimens examined were variable mixtures of calcium and magnesium carbonates with silica, ferric oxide, and aluminum oxide. All were from the Niagara formation. Some proved to be nearly typical dolomites, which contain 45·65 per cent of magnesium carbonate and 54·35 per cent calcium carbonate.

At first a complete analysis of each specimen was made. The general method was to weigh about a grm. of the fine powder in a small beaker; to dissolve carefully (the beaker covered with a watch-glass) in pure dilute hydrochloric acid by gently heating to the boiling-point. The insoluble residue, consisting of silica, was filtered and determined. The amount of silica in these rocks is usually small, and this method, when compared with others, was found to give accurate results. A grm. or two of pure ammonium chloride was added to the filtrate from the silica, and at the boiling temperature it was treated with a small excess of ammonia to precipitate the iron and alumina. The filtrate from these was heated to boiling and precipitated with a N/2 solution of ammonium oxalate, care being taken to avoid much excess of the reagent. The precipitate was allowed to stand eight to twelve hours before filtering. The well-washed precipitate of calcium oxalate, with a small quantity of magnesium oxalate, was dissolved in warm dilute hydrochloric acid, and the solution was rendered alkaline with ammonia; this precipitates the calcium oxalate and leaves the magnesium in solution. This amount of magnesium and the main portion from the first calcium-magnesium precipitate were determined separately and weighed as magnesium pyrophosphate. The carbon dioxide was determined by the Bunsen method. After the complete analysis was made, only the small amount of magnesium, that which precipitates with the calcium, was determined.

#### I. Determinations by C. C. Carhart.

The specimen was nearly a typical dolomite, containing—

|                                                                      |                |
|----------------------------------------------------------------------|----------------|
| CaCO <sub>3</sub> .. .. .                                            | 53·78 per cent |
| MgCO <sub>3</sub> .. .. .                                            | 44·96 "        |
| SiO <sub>2</sub> .. .. .                                             | 0·88 "         |
| Fe <sub>2</sub> O <sub>3</sub> and Al <sub>2</sub> O <sub>3</sub> .. | 0·38 "         |
|                                                                      | 100·00 "       |

The small amount of MgO—i.e., the quantity precipitated with the calcium—was 0·08 per cent.

Eleven other determinations of this amount resulted as follows:—

|           |               |            |               |
|-----------|---------------|------------|---------------|
| 1 .. .. . | 0·26 per cent | 7 .. .. .  | 0·13 per cent |
| 2 .. .. . | 0·16 "        | 8 .. .. .  | 0·11 "        |
| 3 .. .. . | 0·20 "        | 9 .. .. .  | 0·17 "        |
| 4 .. .. . | 0·29 "        | 10 .. .. . | 0·20 "        |
| 5 .. .. . | 0·31 "        | 11 .. .. . | 0·14 "        |
| 6 .. .. . | 0·11 "        |            |               |



This is an average of 0.18 per cent MgO for the twelve determinations, corresponding to 0.378 per cent  $\text{MgCO}_3$ .

The amounts obtained are small, and indicate that a sufficient quantity of ammonium chloride was added after removing the silica, and also that the ammonium oxalate was not added in large excess in the precipitation of the calcium.

#### II. Determinations by F. E. Welstead.

A complete analysis of the specimen gave—

|                                                             |       |          |
|-------------------------------------------------------------|-------|----------|
| $\text{CaCO}_3$ .. .. .                                     | 50.91 | per cent |
| Larger quantity of $\text{MgCO}_3$ .. .. .                  | 42.01 | "        |
| Smaller quantity $\text{MgO}$ , 0.70 per cent =             |       |          |
| $\text{MgCO}_3$ .. .. .                                     | 1.47  | "        |
| $\text{SiO}_2$ .. .. .                                      | 2.08  | "        |
| $\text{Fe}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$ .. .. . | 3.51  | "        |
|                                                             | 99.98 | "        |

2.  $\text{MgO}$  = 0.59 per cent  
3.  $\text{MgO}$  = 0.47 "  
4.  $\text{MgO}$  = 0.75 " } An average of 0.63 per cent.

#### III. Determinations by C. A. Utt.

1. The analysis of the specimen gave—

|                                                             |       |          |
|-------------------------------------------------------------|-------|----------|
| $\text{CaCO}_3$ .. .. .                                     | 52.92 | per cent |
| $\text{MgCO}_3$ .. .. .                                     | 37.56 | "        |
| $\text{MgO}$ , 0.88 = $\text{MgCO}_3$ .. .. .               | 1.84  | "        |
| $\text{Fe}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$ .. .. . | 6.26  | "        |
| $\text{SiO}_2$ .. .. .                                      | 1.44  | "        |

- 100.02 "  
2.  $\text{MgO}$  = 0.96 per cent  
3.  $\text{MgO}$  = 0.15 "  
4.  $\text{MgO}$  = 1.28 "  
5.  $\text{MgO}$  = 0.47 "  
6.  $\text{MgO}$  = 0.228 "

In 1, 2, and 4 of this series, after dissolving the calcium and magnesium oxalates with hydrochloric acid and precipitating with ammonia, the calcium precipitate was at once filtered. It is therefore evident that the calcium was not all precipitated, and afterwards came down and was determined as magnesium. This easily explains the larger amounts obtained in these determinations.

Next such a mixture of pure Iceland spar and dolomite was taken that the calcium carbonate largely predominated.

1. The analysis of the mixture:—

|                                                             |        |          |
|-------------------------------------------------------------|--------|----------|
| $\text{CaCO}_3$ .. .. .                                     | 87.035 | per cent |
| $\text{MgCO}_3$ .. .. .                                     | 10.828 | "        |
| $\text{MgO}$ = 0.47 = $\text{MgCO}_3$ .. .. .               | 0.990  | "        |
| $\text{SiO}_2$ .. .. .                                      | 0.06   | "        |
| $\text{Fe}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$ .. .. . | 1.08   | "        |

- 99.993 "  
2.  $\text{MgO}$  = 0.15 per cent.

Instead of a grm., but 0.2033 grm. of the original substance was used. The small quantity of  $\text{MgO}$  was 0.66 per cent. From the foregoing it appears that it makes no difference if the percentage of magnesium is relatively small or if a small amount of substance is used. About the same quantity of magnesium falls down with the calcium.

#### IV. Determinations by Miss L. B. Safely.

1. The analysis of the substance showed its composition as follows:—

|                                                             |       |          |
|-------------------------------------------------------------|-------|----------|
| $\text{CaCO}_3$ .. .. .                                     | 41.93 | per cent |
| $\text{MgCO}_3$ .. .. .                                     | 42.15 | "        |
| $\text{MgO}$ = 0.81 = $\text{MgCO}_3$ .. .. .               | 1.70  | "        |
| $\text{Fe}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$ .. .. . | 13.62 | "        |
| $\text{SiO}_2$ .. .. .                                      | 0.88  | "        |

- 100.28 "  
2.  $\text{MgO}$  = 0.81 per cent  
3.  $\text{MgO}$  = 0.78 "  
4.  $\text{MgO}$  = 0.80 "  
5.  $\text{MgO}$  = 0.72 "  
6.  $\text{MgO}$  = 1.20 "

In the last case some calcium precipitated with the magnesium, because the calcium precipitate was not allowed to stand.

Next used a mixture of Iceland spar and dolomite in which the calcium carbonate largely predominated.

1.  $\text{MgO}$  = 0.47 per cent  
2.  $\text{MgO}$  = 0.47 "  
3.  $\text{MgO}$  = 0.47 "  
4.  $\text{MgO}$  = 0.54 "

Instead of a grm., used 0.3518 grm. of the original dolomite:—

$\text{MgO}$  = 0.25 per cent.

Again, 0.2133 grm. gave—

$\text{MgO}$  = 0.03 per cent.

Next a specimen of magnesite was used in which the amount of  $\text{CaCO}_3$  was only 1.30 per cent. The small amount of  $\text{MgO}$  was 0.26 per cent.

The conclusion seems to be that the magnesium precipitated with the calcium varies from an almost inappreciable amount to a considerable quantity. It is therefore always better to dissolve the unwashed precipitates of calcium and magnesium in warm hydrochloric acid, then to add ammonia to precipitate the calcium. After standing a suitable time, the calcium may be filtered, and the filtrate can be added to the solution containing the main portion of the magnesium, or the two portions can be separately treated.

Chemical Laboratory, Cornell College,  
January 4, 1904.

### CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS.\*

#### A STUDY OF PRASEODYMIUM: PREPARATION OF PURE MATERIAL—PRASEODYMIUM CITRATE.†

By CHAS. BASKERVILLE and J. W. TURRENTINE.

WE shall adopt, in reporting investigations upon the rare earths, the plan of succinctly stating in an introductory paragraph the facts observed and conclusions arrived at. Those desirous of familiarising themselves with the details may peruse what follows at leisure. Perhaps others may care to pursue a similar course. No doubt a wider dissemination of the actual results arrived at will come about, and the labours of abstractors be lessened and more accurate. Expediency influenced literary style before the Twentieth Century.

**Synopsis.**—This paper contains a description of a novel method for rapidly preparing pure compounds of praseodymium. It depends essentially upon saturating a concentrated water solution of citric acid with ammonium-free praseodymium hydroxide, in which the contaminating lanthanum is below 10 per cent. The solution is heated, whereby a new normal citrate is immediately precipitated and readily washed free from acid. The other procedures giving negative results were precipitations by potassium iodate, fractionating by hydrofluoric acid, gaseous hydrochloric acid, formaldehyde, fusion with potassium pyrosulphate, alkaline hydroxides, digestion in concentrated alkaline solutions, fusion with sodium peroxide, and attempts to form alums.

Fairly complete synopses of the observations leading to the breaking up of Mosander's didymium (*Liebig's Ann. Chem.*, xlv., 125) into several elements have been given in an historical consideration of the rarer earths (*Science*, 1902, N.S., xvii., 772; see also Dennis and Dales, *Journ. Am. Chem. Soc.*, 1902, xxiv., 425). To Dr. Carl Auer, without doubt, belongs the main credit of renewing interest in and stimulating the most modern assaults upon these interesting bodies. The former grew out of his patient research, resulting in the announcement of neo- and praseodymium (*Monatsh. Chem.*, 1885, vi., 477). The latter was

\* This and the following dozen or more papers were presented in abstracts in a Lecture delivered before the New York Section under the title "The Rare Earth Crusade: What it Portends, Scientifically and Technically" (*CHEMICAL NEWS*, lxxxviii., 18).

† Presented at the Pittsburgh Meeting of the American Chemical Society, 1902. From the *Journal of the American Chemical Society*, 1904, xxvi., No. 1.

the outcome of his development and the extensive use of mantles composed of thorium and its co-geners for artificial illumination. The intimate relationship and the reciprocal dependence of pure and applied chemistry could not find a better illustration. The commercial demand for thorium compounds especially has caused extensive mining of and geologic investigations for the better known natural compounds, as cerite and monazite, and utilisation of several others previously regarded as rare curiosities of the mineral cabinet. In the extraction of the useful thorium oxide, there have accumulated tons of crude salt mixtures of the rarer elements biding a time of commercial extraction and utilisation.

The Welsbach Lighting Company, of Gloucester City, N. J., through their late Chemist, the lamented Dr. Waldron Shapleigh, and his long time associate and successor, Dr. H. S. Miner, has aided scientific men frequently in generously loaning preparations extracted and often purified to a high degree. This, and a number of other researches which follow in this series, have been facilitated and rendered possible in certain cases by Mr. Miner's gratuities, and we wish here to express profound thanks.

During the preparation of pure thorium compounds, it was noted (by B., *Journ. Am. Chem. Soc.*, xxiii., 761) that a concentrated solution of citric acid, saturated with thorium hydroxide, precipitated a pure thorium citrate on heating. (By "pure thorium" is meant the "old thorium" without considering the recent researches of Schmidt, the Curies, Crookes, Rutherford, Brauner, Baskerville, and others.) After destruction of the carbonaceous material and re-solution of the thorium oxide, no absorption bands were observable with a strong beam of light passing through 15 c.m. of the concentrated sulphate or chloride. In the preparation of thorium tetrachloride to be used in re-determining the atomic mass of thorium, a slight red in several cases and a green deposit in others (sometimes together) was formed in the hard glass tube several centimetres beyond the boat in which the mixture of carbon and oxide was heated. While the absorption spectrum has proved a most valuable means of investigating certain of the rarer earths, as has been well pointed out, it is not as extremely delicate as one might wish. Boudouard (*Comptes Rendus*, cxxvi., 12) has been able to separate small amounts of neodymium from quite pure yttrium preparations by repeated fractionations when the material had long since failed to show the absorption bands of the former complex. Cleve has observed the same with rather pure samarium (*CHEMICAL NEWS*, ii., 145). It has been learned in this laboratory that when quite pure neodymium and praseodymium oxides are mixed with carbon and heated to a dull red in an atmosphere of chlorine they do not give rise to volatile bodies. However, we have learned that praseodymium chloride, when subjected to a temperature sufficiently high to soften combustion tubing, evolved a greenish body. (This will be taken up in one of the subsequent papers.) Matignon has also observed the same in regard to neodymium, when a pink-coloured deposit is formed in the cooler parts of the tube (*CHEMICAL NEWS*, lxxiv., 97; lxxv., 132; *Comptes Rendus*, cxxiii., 5). From the colour of these bodies, it was immediately assumed that, in spite of the prolonged and painstaking method of preparation, our pure thorium compounds (*loc. cit.*) still retained a trace of the constituents of didymium, which hangs on in other cases. Investigations were at once begun therefore to learn the conduct of these elements with citric acid—the only novel feature in the method of purification not previously tested.

Before giving the result of those experiments, it may be well to state that up to that time the chlorine used had been generated by the action of pure concentrated hydrochloric acid upon re-crystallised and granulated potassium dichromate. Subsequently a cylinder of pure chlorine was substituted, with a complete cessation of the trouble. These red and green deposits were very small, but persisted in forming as long as the chlorine was prepared by the chromate method, and finally proved to be due to chromium.

This was an observation of no little interest, as the chlorine gas was thoroughly scrubbed by passing through four wash-bottles of concentrated pure sulphuric acid (98 per cent) and two columns of beads and one tower of pumice saturated with the same desiccating agent before entering the combustion tubing. One is immediately reminded of Crookes's difficulty in preventing a back flow of mercury vapour in evacuating the bulbs for his beautiful phosphorescence spectra examinations, when he states there is "no use trusting to a solid reagent to absorb a gaseous body" (*CHEMICAL NEWS*, liv., 29).

Praseodymium ammonium nitrate prepared by the method of Auer (*Monatsh. Chem.*, vi., 477) was converted into the hydroxide, and washed by decantation until the ammonium salts were virtually removed. Twenty-five c.c. of a concentrated water solution of citric acid were saturated with the praseodymium hydroxide by shaking in a glass-stoppered bottle; the undissolved hydroxide filtered away, and the green solution heated. Immediately a pale green amorphous precipitate formed, settled upon the bottom of the vessel, was easily filtered, and washed with hot water.

Similar experiments were carried out with neodymium and lanthanum hydroxides. The former is about one-tenth as soluble in citric acid as praseodymium hydroxide, and is not precipitated on boiling, until the solution is very concentrated.

Lanthanum hydroxide is intermediate in its solubility and not precipitated at once on boiling. It requires concentration and continued heat, although both of these substances allow tough, whitish precipitates to form, when a very strong solution is permitted to stand some days at the temperature of the room. Perhaps here we may have to do with such colloidal compounds of the rare metals as mentioned by Delafontaine (*CHEMICAL NEWS*, lxxiii., 284). This point requires investigation, however.

Numerous attempts were made to apply the observations noted to separating lanthanum from the didymiums as they occur in monazite sand and other rare earth minerals, but with little success. The lanthanum apparently causes a supersaturated solution to form, which it is difficult to precipitate by heat; further, when a precipitate forms, it contains all three of the elements\* in all the fractions obtained.

The praseodymium ammonium nitrate contained about 10 per cent of lanthanum. One precipitation, according to the procedure mentioned, was sufficient to give a body absolutely free from lanthanum, as shown by the arc spectrograph made with a concave Rowland's grating, 15,000 lines to the inch and 21 feet diameter.† This was determined frequently with that part of the spectrum which shows the strong lanthanum lines.

*Praseodymium Citrate.*—The body was prepared on different scales, using from a few grms. of praseodymium ammonium nitrate to as much as 4 kilograms., in one place. The cold citrate solution (as much as 5 litres of the solution being used in certain experiments) was placed in a tall cylindrical precipitating jar, and stirred by a water motor while the hydroxide was added to complete saturation, the agitation being continued from two to four hours. The temperature was kept down by placing the jar in running water. The turbid liquid was then quickly filtered by upward suction through an unglazed porcelain pear-shaped bomb. The beautiful, clear, leek-green solution was heated in large Jena glass beakers submerged in baths of boiling water, and filtered through a hot funnel, suction being applied at times, by means of the biscuit-ware mentioned, a most useful piece of apparatus when working on the large scale in the laboratory. The precipitate was readily and quickly washed by boiling water, in which it is insoluble (the washings being kept separate), until the filtrate was no longer acid to litmus.

\* The term "element" is used advisedly here, although there is no doubt of neodymium and praseodymium being "complexes," as will be shown in later papers. At present, however, most chemists look upon these bodies as simple.

† Dr. W. J. Humphreys, of the Rouss Physical Laboratory, University of Virginia, kindly prepared these plates, a discussion of which he will publish in due time.

The precipitate, when dried, was a loose powder, amorphous, of a beautiful pale green colour. On analysis the following results were obtained:—

| Found.        | Calculated for (Pr=140.5)                                      |       |                                                                                   |       |
|---------------|----------------------------------------------------------------|-------|-----------------------------------------------------------------------------------|-------|
|               | Pr(C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> ) <sub>3</sub> |       | Pr(C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> ) <sub>2</sub> .H <sub>2</sub> O. |       |
| Oxide (black) | 48.83                                                          | 48.70 | As PrO <sub>2</sub> .. 52.35                                                      | 49.64 |
|               |                                                                |       | or As Pr <sub>2</sub> O <sub>3</sub> .. 49.90                                     | 48.49 |
| Carbon ..     | —                                                              | —     | 21.05                                                                             | 21.18 |
| Hydrogen ..   | —                                                              | —     | 21.85                                                                             | 20.72 |
|               |                                                                |       | 1.46                                                                              | 1.86  |
|               |                                                                |       | 1.52                                                                              | 1.44  |

**Remarks and Precautions.**

Ammonia prevented the formation of this normal citrate in this manner; hence the necessity for thoroughly washing the hydroxide. The citrate is soluble in ammonium citrate. So far, we have not sought to separate the double salt which is evidently formed.

As is well known, lanthanum hydroxide, and in fact most of these so-called trivalent rare earth hydroxides, combine with the carbon dioxide of the air, whereby a partial solution results, and an increasing turbidity of the wash-water that is decanted is observed, depending upon the poverty of ammonia and its salts. We avoided this by rapid working, making use of the largest unglazed porcelain suction filter made.

The filtrate from the citrate precipitate was concentrated by heat, and successive precipitates formed, and were separated. They were mealy precipitates, which, on drying formed light but tough masses, each succeeding mass being tougher than the preceding and more difficult to disintegrate into a powder. The fifth appeared in the solution as flocculent agglomerations like paper pulp, and, on drying, became hard and leathery. The following precipitates presented the same appearance, and were almost white in colour. The change in form and the disappearance of the green colour were progressive throughout each series that was carried out. The percentage of oxide in the bodies correspondingly decreased from 48.83 per cent to 35.77. The third precipitate gave strong lanthanum lines in the arc spectrum. They were not further investigated, and are hardly worthy of the time necessary in view of the following facts:—

In attempting to purify praseodymium on the large scale by this method, we observed that when the percentage of lanthanum rose above twenty, no success attended our efforts. By the method of Auer (*loc. cit.*) a large proportion of the lanthanum is first crystallised out as double ammonium nitrate. It has been observed by Dennis (*Yourn. Am. Chem. Soc.*, 1897, xix., 800), Bettendorf (*Liebig's Ann. Chem.*, cclvi., 163), and others, that didymium breaks up the more readily, accompanied with less frequent formation of supersaturated solutions, difficult to handle, when lanthanum is present in notable amounts. By this modified Welbach method, praseodymium may be readily separated from neodymium, but it is a more difficult proposition to secure the latter devoid of the former. To separate pure neodymium, Boudouard (*Comptes Rendus*, cxvii., 12; *CHEMICAL NEWS*, lxxvii., 193) and Muthmann (*Ber. d. Chem. Ges.*, xxxi., 1731) advocate crystallising the double potassium sulphate, but this does not leave either one free from lanthanum, unless the material is subjected to a prolonged series of fractional crystallisation, almost exasperating in their consumption of time, as all who have worked with these rare earths know.\* With these facts in mind, it becomes evident at once that a quick method for the separation of 5 to 10 per cent of lanthanum is worth while, and one that separated the praseodymium absolutely pure is most desirable. In fact, one phase most engrossing rare earth work at present has to do with finding such means for the separation of pure compounds.

Our last work was with 7 kilograms. of praseodymium ammonium nitrate free from neodymium, but containing more than 20 per cent of lanthanum. The material had undergone 300 crystallisations and came from about 100 tons of monazite. A water solution was precipitated by

ammonium hydroxide in large glazed earthenware vessel<sup>8</sup> (100-litre capacity), provided with spiral outlets by which, on removing rubber stoppers, the supernatant liquid might be drawn from different heights. The excess of ammonium nitrate was thus removed: by two washings to prevent the attack of platinum (by its decomposition) in which the entire precipitate was ignited in portions. To decrease the percentage of lanthanum present, the method of Auer (*Monatsh. Chem.*, v., 508) proved satisfactory, and is not expensive, viz., the mixed oxide were added to dilute nitric acid until almost saturated, an account being kept of the weight, and then as much more oxide added. The more basic lanthanum forms the nitrate and remains in solution, taking a part of the praseodymium, as might be expected, while the major portion of the latter oxide is undissolved. Lanthanum, of course, is still present in the undissolved portion, but in much smaller percentage. The difference in the affinity, at least towards nitric acid, is not sufficient to serve as a method for bringing about a rapid separation of these bodies in the pure state. The solution was separated by filtration, washed once or twice, and the respective fractions converted into hydroxides of the earths free from ammonia. Citric acid saturated with the hydroxide from the nitrate solution gave no precipitate on boiling, while that from the undissolved oxides immediately gave an excellent separation of the citrate, yielding over a kilogram. of pure praseodymium oxide on ignition. The material is absolutely free from lanthanum, and as pure as that used by Jones (*Am. Chem. Journ.*, 1898, xx., 5, 345) in his atomic weight determination. It may be well to state that Dr. Humphreys made the spectrographs for Dr. Jones and had the plates for comparison. Although chemically pure citric acid free from lead, ammonia free from silica and sulphuric acid, and nitric acid giving no residue on evaporation, and re-distilled water were used, the preparation contains such impurities as would be gathered from the containers used in the process, but they are in such small amounts as not to vitiate the results reported in several of the following papers, and of such a character that they may be readily removed at the stage it is deemed necessary.

This material as a neutral nitrate, examined at various dilutions and different thicknesses, showed an absorption spectrum that is given for the normal so called elementary praseodymium (bands 569, 482, 459, and 443), and was used in the other work reported in this series. The absorption spectrum was examined and photographed with a Steinheil grating (14,438 lines to the inch) spectroscopic, as described by Dennis (*Yourn. Am. Chem. Soc.*, xiv., 415). The instrument is essentially the same, except that it is a size larger and has a quartz-slit. The source of light used at first was a 100-candle power Edison incandescent light with a helixoid filament. Latterly we have substituted a 600-candle power arc light enclosed in a projection lantern which serves to focus the rays upon the slit. The bright lines of carbon and its impurities, once noted, do not interfere with the observations of the bands. The instrument was purchased with a grant from the Baché Fund of the National Academy.

During the progress of this work, the application of other methods for accomplishing the same object were attempted. Separating by potassium iodate, hydrofluoric acid, crystallising a concentrated chloride solution by saturating with gaseous hydrochloric acid, formaldehyde, fusion with potassium pyrosulphate, alkaline hydroxides, digestion of rare earth\* hydroxides in concentrated alkaline solutions, fusion with sodium peroxide, and endeavouring to form alums, were tried, and they either gave negative results or such as did not warrant pursuit.

**Conclusion.**—Pure praseodymium compounds may be prepared by heating a saturated citric acid solution from which praseodymium citrate separates when the percentage of lanthanum is below 10, in a few hours, while by the former methods weeks or even months were required.

\* A paper follows, giving a new method for freeing neodymium of lanthanum.

\* See papers on "Lanthanates," "Neodidymates," "Double Sulphates," &c., following.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.

By CHARLES BASKERVILLE, Ph.D., F.C.S., Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 137).

## LIST OF PROPOSED CHEMICAL ELEMENTS.

NOTE.—The italicised names are elements which have been tried and found wanting; those in small capitals have been verified beyond question as distinct, although in specific cases evidence is had that they are complex. All others uniformly stand before the bar of judgment. The arrangement is chronological. Due to pressure of affairs, it has been quite impossible in some cases to consult the original papers, hence part of the table is composed of second-hand and meagre information. Every source available has been drawn upon, as Venable's "The Elements, Historically Considered" (*Journal. Elisha Mitchell Scientific Society*, 1887, iv., 36), which unfortunately gives no references; Winkler's "The Discovery of New Elements within the last Twenty-five Years" (Lecture before the German Chemical Society, *Smithsonian Report* for 1897, 237); Cleve's "Marignac Memorial Lecture," 1895; "Rise and Fall of the Defunct Elements" (*CHEMICAL NEWS*, 1890, xxii., 208); "List of Elementary Substances announced from 1877 to 1887" (*CHEMICAL NEWS*, lviii., 188), by the lamented H. Carrington Bolton, to whom I cannot too strongly emphasise my indebtedness for his ever-ready help and sympathy. It is my desire to have this as complete and authentic as possible, I therefore beg that all information as to omissions and corrections be forwarded me (Chapel Hill, North Carolina, U.S.A.); it will be gratefully acknowledged.

| Date.                  | Name.                                        | Source.                      | Authority.                                    | Reference.                                                                         | Remarks.                                                                                                                                                |
|------------------------|----------------------------------------------|------------------------------|-----------------------------------------------|------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1777<br>1778           | <i>Terra nobilis.</i><br>MOLYBDENUM.         | Diamonds.<br>Lead ores.      | Tobern Bergman.<br>Scheele.                   | Kong. Vet. Akad. Handl.,<br>247.                                                   | Molybdena meant number of substances carrying lead.<br>Hjelm (1790) ( <i>Ann. de Chim.</i> , 4, 17) isolated the element.                               |
| 1780                   | <i>Hydrosiderum.</i>                         | Cold-short iron.             | Meyer.                                        | Schrift. Ges. Nat. Freunde,<br>Berlin, II., 334; III., 380.                        | Wassereisen, obtained by dissolving crude iron in acids.<br>Klaproth proved it to be iron phosphide.                                                    |
| 1781                   | TUNGSTEN.                                    | Scheelite.                   | Scheele.                                      | Kong. Vet. Akad. Handl.,<br>89.                                                    | Acid separated from scheelite. Bros. D'Elhular proved it to be in wolframite. The two minerals had long been known.                                     |
| 1781<br>1782<br>(1798) | <i>Siderum.</i><br>TELLURIUM.                | Cold-short iron.<br>Native.  | Bergman.<br>von Reichenstein and<br>Klaproth. | Crell's Ann., 1798, 1, 91.                                                         | Proved to be iron phosphide.<br>Regarded aurum paradoxum or metallum problematicum—and still is.                                                        |
| 1784<br>1788           | Saturnum.<br><i>Diamanthspatherde.</i>       | Lead ore.<br>Corundum.       | Monnet.<br>Klaproth.                          | Journ. de Physique, 28.<br>Beschaff. Ges. Nat. Freunde,<br>Berlin, 8, 4.           | Composed of silicon, iron, and aluminum oxides.                                                                                                         |
| 1789                   | URANIUM.                                     | Pitchblende.                 | Klaproth.                                     | Crell's Ann., 2, 387.                                                              | Metal not separated until 1842, then by Peligot ( <i>Ann. de Chim.</i> , 5, 5).                                                                         |
| 1789                   | ZIRCONIUM.                                   | Zircon.                      | Klaproth.                                     | Klaproth's Beitrage, 1, 203;<br>Crell's Ann., I, 7; <i>Ann. de Chim.</i> , 1, 238. | See work of Berlin (1860).                                                                                                                              |
| 1790                   | <i>Australium</i> or Syd-<br>neum.           | Sand.                        | Wedgewood.                                    | Crell's Ann., 1, 40, 103.                                                          | Hatchett showed this Australian sand to contain iron, aluminum, and silicon oxides.                                                                     |
| 1791                   | <i>Menachite.</i>                            | Magnetic Sand.               | McGregor.                                     | Sv. Vet. Akad. Handl., 1794,<br>137; Crell's Ann., 1796, 1, 313.                   | Klaproth four years later discovered titanium ( <i>Beitr.</i> , 1, 233), and in 1797 found it to be identical with menachite ( <i>Beitr.</i> , 2, 236). |
| 1794                   | YTTRIUM.                                     | Gadolinite.                  | Gadolin.                                      | Scherer's Allg. J.<br>Scherer's Allg. S., 4, 312;<br>Gehlen's Allg. J., 1, 445.    | Named by Ekeberg (1799) ( <i>Crell's Ann.</i> , 2, 63).                                                                                                 |
| 1799<br>1800           | <i>Nameless earth.</i><br><i>Augusterde.</i> | Beryl.                       | Fernandez.<br>Trommsdorff.                    | Annalen.                                                                           | Vauquelin showed it to be calcium phosphate, now known as the mineral apatite.                                                                          |
| 1800                   | <i>Andronia.</i>                             | Charcoal and salt-<br>petre. | Winterl.                                      |                                                                                    | Committee of the French Academy showed it was composed of lime, alumina, silica, and iron oxide.                                                        |
| 1800                   | <i>Thelike.</i>                              | Marble.                      | Winterl.                                      |                                                                                    |                                                                                                                                                         |

|                      |                                      |                                            |                                      |                                                                                                                                                                                                                                                                                   |
|----------------------|--------------------------------------|--------------------------------------------|--------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1801<br>1803         | Alkali pneum.<br>Erythronium.        | Borax.<br>Lead ore.                        | Hahneman.<br>del Rio.                | Proved to be borax.<br>Because became red when heated with acids. Later Descotils and del Rio concluded it was impure chromium oxide (Ann. de Chim., 53, 268). See Vanadium.                                                                                                      |
| 1801<br>(1844)       | COLUMBIUM.<br>(Niobium).             | Columbite.                                 | Hatchett.<br>(Rose).                 | After Wollaston (1809) (Phil. Trans., 99, 246) had said columbium and tantalum were the same and it was accepted, effective work was done by H. Rose (Pogg. Ann., 70, 572; 71, 159, and many later), who re-named it niobium.                                                     |
| 1802<br>(1844)       | TANTALUM.                            | Finnish tantalite and ytrotantalite.       | Ekeberg.                             | Important work on this element also done by Berzelius (Pogg. Ann., 4, 6) and H. Rose (Pogg. Ann., 15, 145, &c.). See Columbium.                                                                                                                                                   |
| 1803<br>1804         | Silene.<br>CERIUM.                   | Cerite and ochroiterde.                    | Proust.<br>Berzelius and Heskinger.  | This complex later shown to contain lanthanum, but the name remains for the element.                                                                                                                                                                                              |
| 1805<br>1811         | Niccolanum.<br>Yunonium.             | Cobalt ores.                               | Klaproth.<br>Richter.<br>Thomson.    | Shown to be mixture of nickel, cobalt, arsenic, and iron. Identity with cerium proved by Wollaston.                                                                                                                                                                               |
| 1817                 | Thorine.                             | Gadolinite.                                | Berzelius.                           | Berzelius later (K. Vet. Acad. Handl., II., 4, 184) proved this to be yttrium phosphate.                                                                                                                                                                                          |
| 1818                 | Vestium or Sirium.                   | Nickel ore.                                | Vest.                                | Faraday showed it to be a mixture of nickel, iron, sulphur, and arsenic.                                                                                                                                                                                                          |
| 1818                 | Wodanium.                            | Cobalt ore.                                | Lampadius.                           | Stromeyer showed it consisted of nickel, arsenic, &c. Wodankies, present mineral gersdorffite.                                                                                                                                                                                    |
| 1820                 | Crodonium.                           | Sulphuric acid in crustation.              | Trommsdorff.                         | Discoverer afterwards demonstrated it to be mainly calcium and magnesium salts with copper and iron impurities.                                                                                                                                                                   |
| 1820<br>1821<br>1828 | Aurum millium.<br>Apyre.<br>THORIUM. | Thorite.                                   | Mills.<br>Brugnatelli.<br>Berzelius. | While no doubt a definite element as accepted, there is no question as to its complexity (see Chroustchoff, Schmidt, Curie, Rutherford, Brauner, and Baskerville).                                                                                                                |
| 1828                 | Ruthenium.                           | Platinum ore (Urals).                      | Osann.                               | Not to be confounded with the well-known ruthenium of Claus. Said to be metal of a golden lustre. Osann previously (Pogg. Ann., 13, 287) gave the name to reddish volatile prisms pronounced by Berzelius new (vide Howe's Vice-President Address before this Section, 1900, 92). |
| 1828                 | Pluranium.                           | Platinum ore (Urals).                      | Osann.                               | Shown to be a mixture of oxides of titanium, zirconium, and silicon by Osann (1829) (Pogg. Ann., 15, 158).                                                                                                                                                                        |
| 1828<br>1830         | Polinium.<br>VANADIUM.               | Platinum ore (Urals).<br>Swedish iron ore. | Osann.<br>Sefstrom.                  | Proved to be impure iridium by the discoverer. About same time Wöhler showed identity of this with erythronium (Pogg. Ann., 21, 39).                                                                                                                                              |
| 1836<br>1836         | Donium.<br>Treenium.                 | Aberdeen mineral.                          | Richardson.<br>Boase.                | Identical with glucinum, as shown by Heddle. Identical with donium.                                                                                                                                                                                                               |
| 1839                 | LANTHANUM.                           | Ceria.                                     | Mosander.                            | Much work done by Brauner looking towards its complexity; so far regards it simple.                                                                                                                                                                                               |

(To be continued).

## PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, March 3rd, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

(Concluded from p. 142).

40. "Photochemically Active Chlorine." (A Preliminary Notice). By CHARLES HUTCHENS BURGESS and DAVID LEONARD CHAPMAN.

By taking advantage of several new facts, the authors have succeeded in showing that the activity of a mixture of hydrogen and chlorine depends entirely on the condition of the chlorine. Draper originally maintained that this was the case, but Bunsen and Roscoe were unable to repeat his experiments, and Mellor, accepting the results of Bunsen and Roscoe, has constructed his working hypothesis on the assumption that Draper is wrong. P. V. Bevan (*Phil. Trans.*, 1903, ccii., 71) has quite recently shown that similar mixtures of hydrogen with insolated and unisolated chlorine exposed to light under the same conditions behave somewhat differently, the period of induction of the former being slightly shorter. The work of Bevan shows that before hydrochloric acid can be formed, some change in the chlorine alone must take place, but it may also be necessary for a further change to occur which can only be effected in the presence of hydrogen, or, in terms of this author's hypothesis, it may first be necessary for complex molecules of chlorine and water to be formed, and then for the hydrogen to react with these, forming complexes containing water, chlorine, and hydrogen, both changes requiring a finite time for their completion.

That a saturated solution of chlorine in water can exist either in an active or in an inactive condition is shown in the following way:—A mixture of hydrogen and chlorine contained in a Bunsen and Roscoe's actinometer was exposed to light until the period of induction was passed. It was then vigorously shaken so as to mix the gas and liquid thoroughly. When the mixture was again exposed to light, a second period of induction was observed, which, however, was not so long as the first. A still shorter period of induction was indicated when the mixture had again been shaken, until after repeating the above process several times no difference could be noticed in the rate of combination before and after shaking, thus proving that the liquid in the bulb had become active. The change which precedes the formation of hydrogen chloride undoubtedly extends to the aqueous solution.

It was next shown that a mixture of hydrogen and chlorine which had passed through the period of induction could again be rendered inactive by shaking the mixed gases with either water, hydrochloric acid, hypochlorous acid, or chlorine water, and further that a mixture of hydrogen and chlorine which had been agitated with water showed a much longer period of induction than one which had not been thus treated.

An apparatus was devised into which chlorine could be passed and treated in any desired manner before being mixed with hydrogen. Into this apparatus, chlorine was passed and shaken with water. The chlorine water thus formed was removed, and the gas again shaken with a fresh supply of water, the operation being repeated several times. The chlorine which had been thus treated was then mixed with an equal volume of hydrogen and exposed to light, when the period of induction lasted for over an hour.

In the next experiment, the chlorine was constantly shaken with the liquid contained in the apparatus, and exposed to a strong light in order to render both the liquid and the gas active; the gas was then mixed with hydrogen in the dark and exposed to light. There was no period of induction; combination of hydrogen and chlorine took place immediately, and no variation in the rate of combination could be observed throughout the reaction.

These results indicate (1) that a solution of chlorine exists either in an active or in an inactive state; (2) that the period of induction is entirely due to the condition of the chlorine.

41. "The Union of Hydrogen and Chlorine. VIII. The Action of Temperature on the Period of Induction." By JOSEPH WILLIAM MELLOR.

Measurements of the duration of the period of induction at different temperatures from 3° up to 50° show (1) that the period is shorter the higher the temperature; (2) that above 38°, disturbing effects, probably due to the presence of water vapour, obscure the influence of the increased temperature.

42. "The Union of Hydrogen and Chlorine. IX. Further Experiments on the Action of Light on Chlorine." By JOSEPH WILLIAM MELLOR.

Bevan has recently shown that the activity of chlorine after exposure to sunlight, first observed by Draper, is destroyed by bubbling the chlorine through water, as in Bunsen and Roscoe's experiment. Bevan supposes that the union with hydrogen still proceeds through the intervention of the intermediate compound,  $\text{xCl}_2\cdot\text{yH}_2\text{O}\cdot\text{zH}_2$ , as suggested in the author's paper (*Trans.*, 1902, lxxxi., 1280). Two bulbs of ordinary moist chlorine were exposed to sunlight for ten minutes, and afterwards mixed with moist hydrogen. The duration of the period of induction with this mixture was compared with that of a similar mixture made in darkness. Two more bulbs of chlorine, dried with phosphoric oxide, were treated in exactly the same way. The periods of induction were relatively as follows, the expansion being corrected for the Budde effect:—

| Standard. | Moist. | Dry. |
|-----------|--------|------|
| I         | 0.5    | 1.0  |
| I         | 0.3    | 0.9  |

The results show that the greater chemical activity of insolated chlorine is intimately associated with the presence of steam. The alternative hypothesis, put forward in the above-cited paper, is just as satisfactory as the intermediate compound theory.

43. "Additive Compounds of Unsaturated Cyclic Ketones with Hydrogen Cyanide." By ARCHIE CECIL OSBORNE HANN and ARTHUR LAPWORTH.

The applicability of the general method for bringing about the addition of hydrogen cyanide to the ethylenic union in a 'unsaturated ketones, nitriles, &c.', recently worked out by one of us (*Trans.*, 1903, lxxxiii., 999), is being systematically investigated, particularly with regard to the open chain and cyclic ketones and aldehydes.

Carvone, in presence of potassium cyanide, unites with one molecular proportion of hydrogen cyanide in the cold. The product, which is formed in nearly quantitative amount, is a *nitrile*,  $\text{C}_{10}\text{H}_{15}\text{O}\cdot\text{CN}$ , melting at 93.5–94.5°, which, on hydrolysis, yields two isomeric unsaturated acids,  $\text{C}_{10}\text{H}_{13}\text{O}\cdot\text{CO}_2\text{H}$ , melting at 137° and 96–97°. The foregoing nitrile gives a *cyanohydrin*,  $\text{C}\cdot\text{C}_{10}\text{H}_{13}(\text{OH})\cdot\text{CN}$ , melting at 106–108°, which is easily hydrolysed, forming the acids,  $\text{C}_{12}\text{H}_{17}\text{O}_3\text{N}$  and  $\text{C}_{12}\text{H}_{15}\text{O}_4$ , which are possibly mixtures of isomerides.

Pulegone behaves somewhat similarly; the first additive product is the *nitrile*,  $\text{C}_{10}\text{H}_{17}\text{O}\cdot\text{CN}$ , which melts at 160.5°.

A number of other derivatives of the foregoing compounds have been obtained, and will be described in due course.

44. "The Formation of Periodides in Organic Solvents." By HARRY MEDFORTH DAWSON.

The formation of potassium periodides in various organic solvents has been studied by determining the composition of such solutions when saturated either with potassium iodide or with iodine.

The aromatic nitro-compounds investigated, such as nitrobenzene, *o*-nitrotoluene, *m*-nitrotoluene, and *o*-nitroanisole, show a very similar behaviour, and under conditions favourable to the formation of the highest possible periodide, namely, saturation of the solution with regard to

iodine, the enneaiodide,  $KI_9$ , is the essential component of the solutions. The composition of the solutions saturated with respect to potassium iodide indicates that increasing concentration favours the formation of periodides of gradually increasing complexity.

By a method devised for the study of solid aromatic nitro-compounds, it is shown that these exert an influence on the formation of soluble periodides which is in many respects similar to that of nitrobenzene.

Other solvents, including nitromethane, nitropentane, ethyl alcohol, propionitrile, ethyl acetate, ethyl bromide, and isobutyl alcohol, have also been investigated.

45. "The Action of Sodium Hypochlorite on the Aromatic Sulphonamides." By HENRY STANLEY RAPER, JOHN THOMAS THOMPSON, and JULIUS BEREND COHEN.

The authors have continued the investigation described in the preliminary notice published in the *Proceedings* (1901, xvii., 262), and have added the following facts:—

*Benzenesulphon-o-toluidide* (m. p. 123–124°), when dissolved in acetic acid and treated with sodium hypochlorite, yields the *chloroamide*,  $C_7H_7 \cdot NCl \cdot SO_2 \cdot C_6H_5$ , melting at 99–100°; this product, on heating with acetic acid, gives *benzenesulphon-5-chloro-o-toluidide* (m. p. 124–125°). *Benzenesulphon-m-toluidide* (m. p. 95°) gives a sodium compound,  $(C_6H_5Cl)(CH_3) \cdot NNa \cdot SO_2 \cdot C_6H_5$ , melting at 275–280°, which is decomposed by acetic acid, and yields *benzenesulphon-6-chloro-m-toluidide*. With an excess of hypochlorite, *benzenesulphon-2:6-dichloro-m-toluidide* (m. p. 114°) is formed. *Benzenesulphon-p-toluidide* forms *benzenesulphon-3-chloro-p-toluidide* (m. p. 110°).

*Benzenesulphon-4-m-xylylide* (m. p. 124–125°) gives *benzenesulphon-5-chloro-4-m-xylylide* (m. p. 148–149°) when heated with the hypochlorite solution at 50°.

*Benzenesulphon-a-naphthalide* (m. p. 168°) is quite unaffected by the hypochlorite even on warming. *Benzenesulphon-1-naphthalide* (m. p. 97–98°) forms a sodium compound, which, when decomposed with acid, gives *benzenesulphon-1-chloro-8-naphthalide* (m. p. 130–131°).

Chattaway and Orton have shown that by the action of sodium hypochlorite on the acyl derivatives of the aromatic amino-compounds, the halogen in the first instance enters the ring in the para-position with respect to the amino-group, and then selects the ortho-position. The orienting effect of the sulphonic group is different. The foregoing results show that in the first instance the halogen seeks the ortho-position with respect to the amino-group. If a methyl group is present and if the para-position to the amino-group is free, the conditions are modified, and the halogen enters either the ortho-position to the methyl radicle or the para-position to the amino-group. It is interesting to note that the action of the hypochlorite is retarded in the *o*-substituted sulphonamides; those of *o*-toluidine and *xylydine* react with difficulty, whilst that of *a*-naphthylamine, which may be regarded as an ortho-substituted compound, is quite unchanged.

#### PHYSICAL SOCIETY.

Ordinary Meeting, March 11th, 1904.

Dr. R. T. GLAZEBROOK, F.R.S., President, in the Chair.

A PAPER on "The Whirling and Transverse Vibrations of Shafts" was read by Dr. CHREE.

The "whirling" of rotating shafts was treated theoretically by Prof. Greenhill in the *Proceedings of the Mechanical Engineers*, 1883, and by Prof. Dunkerley in *Phil Trans.*, Sect. A, 1894. The latter considered the subject in great detail. He made a number of calculations based on two methods—one the same as Prof. Greenhill's, the other due apparently to Prof. Osborne Reynolds—and he compared them with experiments made on a model shaft and pulleys. The second method was employed in all cases where the shaft was loaded; it is dependent on a hypothesis which, as presented by Dunkerley, is largely empirical. The present

paper proceeds from a different standpoint, based on the true nature of the relationship between whirling and transverse vibrations, which seems to have escaped previous notice. It shows how the mathematical results obtained by Prof. Dunkerley can be derived by a less cumbersome treatment, and how in many instances they admit of great simplification, without sensible loss of accuracy. It is also shown how loaded shafts can be dealt with, without recourse to Dunkerley's hypothesis; at the same time some light is thrown on the relation of this hypothesis to theory.

Six main cases are considered, in which the shaft is variously supported; in some of them numerical results are deduced for comparison with Dunkerley's experiments. Some data are given for the stresses due to "centrifugal forces" answering to the velocities at which whirling should appear, and attention is thus called to the necessity of taking these stresses into account when considering the question of safety.

In the main part of the paper results are quoted with only a general indication of the mathematical methods employed; but these methods are illustrated in a short appendix.

The CHAIRMAN said there was one point he would like to bring out in connection with Dr. Chree's paper. The author had referred to a theorem due to Lord Rayleigh: a theorem which he knew well in its application to sound, although he had never seen it employed in connection with rotating shafts. The theorem is that in any vibrating system the period of natural vibrations is a stationary period, so that the application of any small external force produces only a second-order change in the period. By use of this theorem it is possible to assume a definite law and calculate periods with sufficient accuracy by working with expressions much less complicated than would otherwise be the case.

A paper entitled "Notes on Non-homocentric Pencils, and the Shadows Produced by them. Part II. Shadows Produced by Axially Symmetrical Pencils possessing Spherical Aberration," was read by Mr. W. BENNETT.

This paper deals with the shadows obtained by interposing a straight wire near the focus of a pencil proceeding from a lens or mirror uncorrected for spherical aberration. A method is described for drawing sections of the wave-front in the neighbourhood of the focus. A simple physical explanation of the shadows is given by means of the wave-fronts; it is shown that the real shadow consists in general of two branches, one closed and the other open, with a  $\phi$ -form as a symmetrical special case. The equations of the wave-front are worked out for the special case of the reflection of a plane wave at a spherical mirror, and a method of drawing the shadow-forms is described. The equations to the shadows for this case are given in polar and Cartesian forms, and discussed in detail. The paper concludes with an account of the methods adopted for obtaining photographic records, and of a method for viewing the forms directly by replacing the luminous point by the eye. Three experiments were shown to illustrate this last point, as well as a number of the photographs obtained.

Dr. W. WATSON expressed his interest in the paper, and said that the diagrams shown proved that when dealing with problems similar to those considered in the paper, it was better to deal with wave-fronts rather than with rays. In his opinion, the subject of aberrations in lenses was one which should be more frequently studied by students. It was possible to illustrate many important facts by the use of cheap and simple apparatus.

The CHAIRMAN supported Dr. Watson in his remarks with reference to the advantages of dealing with wave-fronts instead of rays in the consideration of optical problems. He impressed upon the meeting the necessity of a more careful study of aberrations and image-formations, stating that papers similar to that read by Mr. Bennett could not fail to be of practical importance in the optical industry.



Messrs. W. G. PYE and Co., of Cambridge, exhibited a large quantity of apparatus manufactured at their works. The exhibit included:—

- A new Compound Reading-Microscope, capable of being used in four positions.
- A large Combination Spectrometer and Spectroscope on somewhat new lines, all motions and fittings arranged geometrically.
- A large Cathetometer.
- Tudsbury's Pneumatic Influence Machine, working in compressed CO<sub>2</sub> giving a thick 6-inch spark.

## NOTICES OF BOOKS

*Microscopic Analysis of Metals.* By FLORIS OSMOND. Edited by J. E. STEAD, F.R.S., F.I.C. With 100 Photographic Illustrations and Two Folding Diagrams. London: Charles Griffin and Co., Ltd. 1904. Pp. 178.

METALLOGRAPHY, or the description of the structure of metals and their alloys, owes much to the method of "polish attack" devised by the author, and combined with polishing in bas-relief and chemical attack, furnishes reactions by which the primary constituents of carbon steels may be defined; it is to this latter object that this volume is principally devoted.

The constituents of carbon steels, apart from the slag and the graphite, are six in number, though further research may reveal others. The chief constituent is the iron itself, and considered structurally it is called "ferrite"; the second constituent is carbide of iron, and is called "cementite," and as it can be obtained in two distinct forms, the mixture is also given another name, viz., "pearlite"; the other of these forms is called "sorbite," after the first pioneer in metallography.

The fourth constituent is obtained by quick quenching in cold water from a temperature a little higher than the maximum temperature of the critical interval, and is called "martensite"; the fifth constituent has been obtained by quenching steels during the critical interval, Ar<sub>1</sub>,<sub>2</sub>,<sub>3</sub>; that is to say, during the course of transformation, and is called "troostite." The sixth and last constituent is called "austenite"; this is obtained by heating the steel to above 1000°, and quenching in a bath at 0° C., or a little below; the proportion of carbon must exceed 1·10 per cent.

After fully describing these constituents and the manner of their occurrence, the author next proceeds to their micrographic identification; then follows a detailed examination of selected steels.

The final chapter deals with the theoretical and practical conclusions to be drawn from the foregoing ones. The principal theoretical conclusion was that the assimilation of quenched steel to solid solutions was placed beyond all doubt, and most convincing examples of the assimilations of solid solutions to liquid solutions were found. Practically it has been seen that the principal circumstances of the calorific treatment of steels reveal themselves in the variations of structure, with a precision that the mere inspection of fractures is far from furnishing, but the different aspects of the structure have not yet been correlated with the corresponding mechanical properties.

The practical application of metallography to metallurgists and engineers requires, therefore, a preparatory study for each metal, but we have no doubt that rapid progress will be made in this direction, so that it will be easy to determine the calorific treatment undergone by a finished article, and to see whether or not it has conformed to specified rules.

We must give a word of praise to the illustrations, which form specimens of microphotography of a high order.

The book is well printed and arranged, and is a useful contribution to metallurgical literature. There is an index.

*Property in Trade Marks*; being a Manual, written without the use of Legal Phraseology, on Trade Marks and their Protection. By BREWER and SON. Second Edition. London: Taylor and Francis. 1904. Pp. 134.

THE ordinary books dealing with the subject of trade marks are couched in such legal phraseology that only the specialists who devote themselves to this subject are at all likely to follow their meaning.

It is to enable the users and prospective users of trade marks to gain a practical knowledge of the somewhat intricate law governing this property that this book has been written.

In addition to the general revision of this edition, two important sections have been added, comprising about sixty extra pages of information. (1) A copy of the official alphabetical list of goods, with numbers of the classes in which such goods have been placed; and (2) a chapter dealing with the danger incurred by a trader in registering his trade mark for a whole class, when he only uses it on some of the goods contained in that class.

Being a "law book" this volume is very probably open to criticism, but such criticism is not within our province; we therefore merely point out that it may be found of use, not only by traders, but also by patent agents and solicitors who have occasionally to attend to such matters.

*Introduction to the Study of Physical Chemistry.* By Sir WILLIAM RAMSAY, K.C.B., F.R.S. London: Longmans, Green, and Co. 1904.

THIS small volume is the first of a series of manuals devoted to the study of different branches of physical chemistry, which are to be published at intervals during the next few months. The editor of the series, Sir William Ramsay, has written this general historical introduction to the volumes, and the writing of the others has in every case been entrusted to authors who are specially qualified to write authoritatively on the separate branches. The plan of issuing manuals rather than one single text-book on a subject which is still in a state of peculiarly rapid development, has many obvious advantages to recommend it, the chief being the ease with which the parts dealing with those individual branches in which more rapid progress has been made can be brought up to date. This volume, in which the treatment is necessarily of a somewhat elementary nature, is eminently suited for being put into the hands of students to lay a firm foundation for the subsequent more detailed study of physical chemistry.

*Praktische Übungen zur Einführung in die Chemie.* ("A Laboratory Outline of General Chemistry"). By Dr. ALEXANDER SMITH. Translated into German from the Second American Edition by Prof. Dr. F. HABER and Dr. M. STOECKER. Karlsruhe: G. Braun. 1904.

IN the preface to the German edition of Dr. Smith's "Laboratory Outline of General Chemistry" the translators pay a generous tribute to the American method of imparting elementary instruction in chemistry to beginners, which according to them is far superior to the German method. This useful practical book, which represents the experience of an admirable teacher, will be of great value in calling the attention of German professors to the American method, which lays great stress on the importance of practical work as a foundation for theoretical instruction. The German edition faithfully preserves the characteristics of the original, which met with a deservedly favourable reception.

*Beiträge zur Chemischen Physiologie und Pathologie.* ("Contributions to Chemical Physiology and Pathology"). Edited by FRANZ HOFMEISTER. IV. Band, 12 Heft. Braunschweig: Friedrich Vieweg und Sohn. 1903.

THE last number of the fourth volume of these *Beiträge* contains only four papers. Of these the third, by Dr.

Leopold Moll, on the artificial conversion of albumin into globulin, appears to be the most important. In it are established some noteworthy relations between the carbonates of the alkalis and ammonium salts as regards their effects upon albumin, on heating to 60°, and the whole article will be found of great interest by physiological chemists. This issue contains the index to the fourth volume, and it is only necessary to read down the titles of some of the papers in order to see how the opportunity afforded by these *Beiträge* of making public the results of many important researches has been appreciated by numbers of distinguished workers in the domain of physiological and pathological chemistry.

*Traité d'Analyse des Substances Minérales. Tome Second. Métaalloïdes.* ("Treatise on the Analysis of Mineral Substances. Vol. II. Metalloids"). By ADOLPHE CARNOT. Paris: Vve. Ch. Dunod. 1904.

The second volume of this treatise on the analysis of mineral substances is devoted to the study of the metalloids. Under the heading of each element a short description is given of its physical and chemical properties and those of its more important compounds, together with methods of determination. No detail of description is overlooked, and all the processes seem thoroughly practical, so that excellent results should be obtained if the clear and adequate directions are carefully followed. Some of the rarer and more recently discovered elements are included, such as titanium, vanadium, molybdenum, &c., which are chemically analogous to the metalloids. Though written from the point of view of pure chemistry, the book contains much that is of importance in the regions of various special industries, and all classes of chemists will be able to learn from its excellent descriptions of the simplest, quickest, and most accurate methods for the quantitative analysis of compounds of the metalloids.

*Recherches sur une Propriété Nouvelle de la Matière.* (Researches on a New Property of Matter"). By M. HENRI BECQUEREL. Paris: Typographie de Firmin-Didot et Cie. 1903.

THIS volume on radio-activity is divided into two parts; the first deals with the author's discovery of the radio-activity of uranium, and his investigation of the physical character of the newly found radiation; though this part is in effect an abstract of papers originally published six or seven years ago, the various observations then made are discussed in full detail in order to show better the successive stages in the progress of our knowledge of the subject. The second part contains an account of the author's work since the discovery by the Curies in 1898 of other substances exhibiting the same phenomena in a far higher degree. Since the monograph aims only at giving a full account of the author's own work, that of the Curies and others is not described as fully as would be necessary in a complete treatise, though it is by no means passed over or minimised. The volume contains some most interesting plates, and a bibliography brought up to September, 1903.

*Up to Date Tables of Imperial, Metric, Indian, and Colonial Weights and Measures.* Simple Suggestions for Metric Adoption; Imperial Decimal Coinage, Foreign, Indian, and Colonial Moneys; Standard Time, Mensuration, Interest; Specially prepared Maps; Metric Measurements applied to Out-door Games. By ALFRED J. MARTIN, Fellow of the Surveyors' Institute, England. London: T. Fisher Unwin.

THE author has brought together a mass of matter connected with the increasingly important subject of the introduction of the metric system, and many arguments are used in favour of its speedy adoption in weights, measures, and coinage, and suggestions are made in view of minimising the temporary inconvenience that must necessarily at first accompany the proposed change.

The author deals in a minute and thorough manner with the effects that the proposed alteration would have upon various trades and professions, and strongly emphasises the absurdity of continuing our present system and the advantages that would follow the change.

The book includes also much information only indirectly connected with the subject, such as examples on the use of logarithms, the Morse code, astronomical symbols, signs of the Zodiac, &c.

Supplementing the above comprehensive work the author has issued a small fifty page penny "Table book" for use in schools, showing how decimals can be taught at a very early age, and containing many original ideas and hints upon the introductions of the elementary principles of the subject. Both books are well printed and of handy size, and will be found of value to both commercial and professional men.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

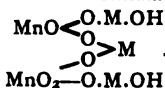
*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxviii., No. 8, February 22, 1904.

**Direct Hydrogenation of Aniline. Synthesis of Cyclohexylamine and two other New Amines.**—Paul Sabatier and J. B. Senderens.—The general method of direct hydrogenation by reduced nickel can be applied to aniline. If aniline vapours containing excess of hydrogen are passed over nickel at a temperature of about 190° an intense absorption of gas takes place, with evolution of ammonia. An almost colourless liquid condenses with a pronounced ammoniacal odour. This liquid contains, besides a small quantity of non-transformed aniline, three amines present in almost equal quantities:—(1) Cyclohexylamine,  $C_6H_{11}NH_2$ , boiling at 134°; (2) dicyclohexylamine,  $(C_6H_{11})_2NH$ , boiling at 250°; (3) cyclohexylaniline,  $C_6H_5NHC_6H_{11}$ , boiling at 275°.

**Experimental Researches on Distillation.**—Eug. Charabot and J. Rocherolles.—Unsuitable for abstraction.

**Alkaline Earth Mangani-manganates.**—V. Auger and M. Billy.—The authors re-investigate the alkaline earth manganates, and find—(1) That none of the products hitherto obtained by the recognised methods possess the formulæ of manganates, but that all contain a less quantity

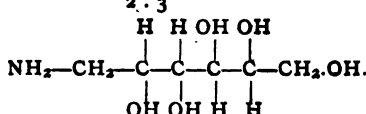
of oxygen than that demanded by the formula  $MMnO_4$ ; (2) that Dulaurier's calcium manganate is only a mixture of lime, manganese dioxide, and calcium manganite. The authors obtain the above products as pure as possible by melting together, at temperatures varying from 180° to 250°, mixtures of potassium permanganate, the alkaline earth base, and a fusible mixture of alkaline nitrates. Oxygen is evolved, and a green mass obtained, which, when treated with suitable solvents, leaves a blue-green powder, insoluble in water, and which, on analysis, is found to possess the formula  $Mn_2O_8M_3H_2O$ , which can be developed into the structural formula—



This can be considered as a compound of a manganite and a manganate, i.e., a mangani-manganate. The authors specially examine barium mangani-manganate.

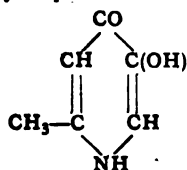
**Action of Carbon Dioxide on Solutions of Sodium Nitrite.**—Louis Meunier.—The author proves that in Marie and Marquis's experiments on the action of carbon dioxide on sodium nitrite, the large quantity of nitrous acid set free is exclusively due to the presence of potassium iodide.

**Mannamine: a New Base derived from Mannose.**—E. Roux.—The author finds that the reduction of mannose oxime gives a new base (mannamine), which represents amino-1-hexanepentol-4.5.6:—

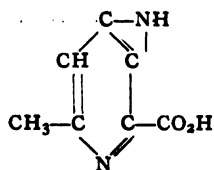


He also describes the properties of the new base and certain of its salts.

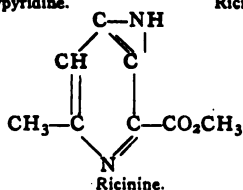
**Ricinine.**—L. Maquenne and L. Philippe.—The reactions given by ricinine, which the authors describe, show that ricinic acid is probably the carboxyl derivative of an iminomethylpyridine. From this they evolve the formulæ of ricinine and the products of decomposition to be cyclic products as follows:—



Methyloxy-pyridine.



Ricininic acid.



Ricinine.

**Inversion of Sugar.**—L. Lindet.—The author performs a series of experiments which control and generalise the inversion of sugar. He finds that when sugar solutions are heated in metallic vessels, the hydrates play an important part in the inversions. If the liquid is aerated, inversion is more active than if the liquid has been previously boiled. An aluminium vessel gives more or less strong inversion, because the sides become covered with alumina, which is a very active agent.

**Simultaneous Existence in Living Cells of Oxidising and Reducing Diastases.**—E. Pozzi-Escot.

## MISCELLANEOUS.

**American Electro-chemical Society.**—The fifth meeting of the American Electro-chemical Society will be held at Washington on April 7th, 8th, and 9th, 1904. The meetings will be held at the Columbian University. Thursday and Friday afternoons will be devoted to visits to scientific laboratories, Government Institutions, and various points of interest in and about Washington; on Thursday evening the Presidential Address will be delivered, and will be followed by a Smoking Concert; on Friday evening there will be a Subscription Banquet. A number of papers are announced to be read, and others are expected, and the attention of members is called to the A. B. Frenzel Prize of 250 dollars, the conditions of which are stated on p. 10, vol. iii., of the *Transactions*.

**The "Bastian" Mercury Vapour Lamp.**—On Saturday afternoon last, March 19th, a private exhibition was given at the offices of Messrs. Rumney and Rumney, 39, Victoria Street, Westminster, of the new "Bastian" Mercury Vapour Lamp. The light given by this lamp closely resembles that of the moon, and will no doubt be well adapted for street lighting; we do not consider it would be a comfortable light for interiors as it is too

"ghostly" in its effects. The light is produced by means of a long arc passing through an exhausted glass tube saturated with mercury vapour; in this respect it resembles other mercury vapour lamps, but it has this advantage, that though the voltage may vary as much as 50 per cent, the lamp burns steadily on, only the quantity of light is reduced as the length of the arc decreases, the quality remaining the same. The "Bastian" lamp is at present adapted for continuous currents only, but we are informed that it is expected before long to get them to work on an alternating current. It is extremely simple in construction, and there is nothing to get out of order, no filament to break, and no carbons to burn up, while the efficiency claimed is 2½ candle-power per watt; each lamp requires anything from 40 to 60 volts and about 0.7 ampère, or the same as an incandescent lamp of 8-candle power, while the light given is no less than 80-candle power, or ten times as much. The colour difficulty, owing to the absence of red rays, is claimed to have been overcome (at a slightly increased cost) by one of the numerous patents taken out by the inventors.

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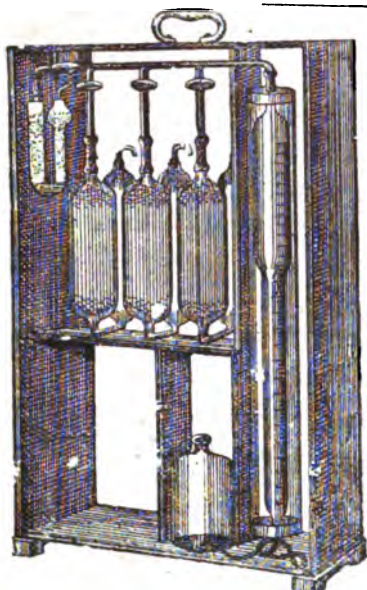
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## CONTENTS.

| ARTICLES:—                                                                                                               | PAGE |
|--------------------------------------------------------------------------------------------------------------------------|------|
| The Existence of Hydrates in Concentrated Aqueous Solutions of Electrolytes, by Harry C. Jones and Frederick H. Getman.. | 157  |
| Contributions to the Chemistry of the Rare Earths, by Chas. Baskerville and Roston Stevenson.....                        | 158  |
| New Forms of Pipettes, by B. M. Mukerjee.....                                                                            | 161  |
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S.....                                          | 162  |
| Eleventh Annual Report of the Committee on Atomic Weights—Determinations Published during 1903, by F. W. Clarke.....     | 164  |
| The Specific Heats of Metals and the Relation of Specific Heat to Atomic Weight, by W. A. Tilden, D.Sc., F.R.S.....      | 165  |
| PROCEEDINGS OF SOCIETIES—                                                                                                |      |
| THE FARADAY SOCIETY.....                                                                                                 | 165  |
| NOTICES OF BOOKS.....                                                                                                    | 166  |
| CORRESPONDENCE.....                                                                                                      | 167  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES.....                                                                               | 167  |
| MISCELLANEOUS.....                                                                                                       | 161  |

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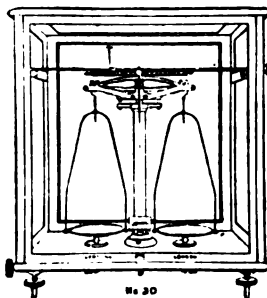
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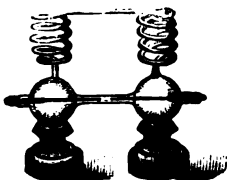
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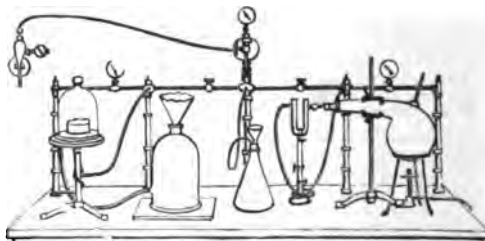
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2314.

## THE EXISTENCE OF HYDRATES IN CONCENTRATED AQUEOUS SOLUTIONS OF ELECTROLYTES.

By HARRY C. JONES

and  
FREDERICK H. GETMAN (Carnegie Research Assistant).

IN our paper published in the Ostwald "Jubelband" (*Zeit. Phys. Chem.*, 1903, xlv., 244), we gave considerable data which seemed to point to the correctness of the suggestion made three years before by Jones and Chambers (*Am. Chem. Journ.*, 1900, xxiii., 89), that in concentrated solutions of electrolytes the dissolved substance is combined with a part of the water forming *hydrates*. This suggestion was originally put forward by Jones and Chambers to account for the fact that such solutions of electrolytes give abnormally great lowerings of the freezing-point of water.

When the work recorded in the Ostwald "Jubelband" was finished, we had studied *thirty-four* compounds—acids, bases, and salts—and had found, in practically every case, abnormally great lowering of the freezing-point of water; if we take as the normal lowering that calculated from the dissociation. We also pointed out (*Zeit. Phys. Chem.*, 1903, xlv., 286) that such solutions of electrolytes give abnormally great rise in the boiling-point of the solvent, and further, that the *minimum in the boiling-point curve* was at a *greater concentration* than in the freezing-point curve. This was in accord with our suggestion of the existence of hydrates in such solutions, since the hydrates formed would be less stable at the higher temperatures, and therefore greater concentration would be necessary for their formation in quantity or complexity sufficient to change the direction of the curve.

Since the above work was done, fifteen other salts have been studied in this connection. They are the chlorides, nitrates, and sulphates of manganese, nickel, cobalt, copper, and aluminium. All of these substances show abnormally great lowerings of the freezing-point of water, except two or three sulphates, which we proved were polymerised in such solutions. Aluminium chloride and nitrate gave *very great* lowerings of the freezing-point of water—the former giving in the most concentrated solution studied, a lowering which was *nearly five times as great as the calculated value*. These results will be published and discussed in full in the April number of the *American Chemical Journal*. Most of these substances also show minima in the freezing-point curves, but this is of subordinate importance.

The point upon which we wish to lay stress in this brief communication is the *relation between the water of crystallisation of a compound and the magnitude of the freezing-point lowering in concentrated solutions*. If electrolytes form hydrates in concentrated solutions, it is only reasonable to expect that, of correlated substances, those that crystallise with the most water will be the substance that will combine with most water in such solutions, will show the greatest amount of hydration. This can be readily tested, since those compounds which, in solution, combine with the greatest amount of water would be the compounds that would produce the greatest lowering of the freezing-point. This is obvious, since the water that is combined with the dissolved substance no longer plays the rôle of solvent, and is removed from the field of action as far as freezing-point lowering is concerned. That such a relation, in general, exists we shall show in our next communication in the *American Chemical Journal*. It can, however, be seen for the chlorides of the alkalis and alkaline earths (*Zeit. Phys. Chem.*, 1903, xlv., 281, Fig. 3), that they stand in the same relation with respect to the

amount to which they lower the freezing-point of water in concentrated solutions, and with respect to the water of crystallisation. The three alkali chlorides, sodium, ammonium, and potassium, have no water of crystallisation, and all lower the freezing-point of water to about the same extent. Lithium chloride, which crystallises at low temperatures with two molecules of water, gives considerably greater lowering of the freezing-point than the alkali chlorides. When we turn to the chloride of the alkaline earths, we come first to barium chloride, which has, under normal conditions, two molecules of water. It gives much greater lowering than even lithium chloride. This, however, is just what is to be expected, since barium chloride crystallises with water under conditions where lithium chloride crystallises with no water. Further, it is a ternary electrolyte, while the chlorides of the alkalis are all binary electrolytes. Barium chloride could not be studied at any great concentration on account of its limited solubility. Strontium chloride and calcium chloride each crystallises with six molecules of water. They show considerably greater lowering than barium chloride.

If we turn to the bromides of the alkaline earths, exactly the same relations appear as with the chlorides, as is evident from the work of Jones and Chambers (*Am. Chem. Journ.*, 1900, xxiii., 96). Comparing Figs. 1 and 2 (*Ibid.*), we find that magnesium chloride, with six molecules of water of crystallisation, gives greater lowering than calcium chloride with six molecules of water, and this in turn gives greater lowering than strontium chloride with six molecules of water. Strontium chloride gives greater lowering than barium chloride with two molecules of water.

Turning to the bromides, magnesium bromide with six molecules of water gives greater lowering than calcium bromide with six molecules of water. Calcium bromide, in turn, gives greater lowering than strontium bromide, which also has six molecules of water of crystallisation. Barium bromide with two molecules gives less lowering than even strontium bromide. The relations between the chlorides and bromides are thus perfectly satisfactory.

If we turn to the nitrates of the alkalis (*Zeit. Phys. Chem.*, 1903, xlv., 281, Fig. 4), we find that lithium nitrate, with two and one-half molecules of water of crystallisation, gives much greater lowering than the nitrates of sodium, ammonium, and potassium, which have no water of crystallisation, and which give about the same lowering of the freezing-point of water.

When we turn to the sulphates of the alkalis, they all give about the same lowerings of the freezing-point, and only sodium sulphate crystallises with water of crystallisation. The amount of water with which sodium sulphate crystallises is a function of the conditions under which it crystallises, and from the nature of the resulting compounds it is questionable whether it holds any water in stable equilibrium.

The carbonates of the alkalis present the only possible exception to our generalisation of the thirty-eight substances that we have thus far studied that have water of crystallisation. Sodium carbonate under ordinary conditions crystallises with more water than potassium carbonate, and gives somewhat smaller lowering of the freezing-point of water. Sodium carbonate, however, readily loses a part of its water of crystallisation when it crystallises with ten molecules of water; and further, can be readily obtained from solution, by varying the conditions under which it crystallises, with seven molecules of water, with five molecules of water, with two molecules of water, and with one molecule of water. The apparent exception presented by these two carbonates is, then, not as well defined as might at first appear. A large number of similar relations exist among the fifteen substances that we have studied since our paper was forwarded to the Ostwald "Jubelband," but these will be seen when our paper is published in the *American Chemical Journal* in April.

It may be said, in general, that of the thirty-eight compounds studied containing water of crystallisation, there is

scarcely a well defined exception to the generalisation, that the molecular lowering of the freezing-point, in concentrated solutions of substances that are closely related chemically, is a function of the number of molecules of water with which the substances crystallise.

We regard this relation as a strong argument in favour of our theory of the existence of hydrates in concentrated solutions of electrolytes. It is well known that many non-electrolytes also give abnormally great lowering of the freezing-point of water in concentrated aqueous solutions. This is probably due to the same cause. It is probable that such substances also form hydrates in concentrated solutions. We shall take this problem up systematically in the near future.

The theory of the existence of hydrates in concentrated aqueous solutions is probably a general theory of such solutions. Of the forty-nine substances that we have thus far studied, all except a few sulphates, which we have proved undergo polymerisation in such solutions, show abnormal freezing-point lowerings in concentrated solutions. Some show a value only a little greater than the value calculated from their dissociation. These are the substances that crystallise with no water of crystallisation, or with very little water of crystallisation. Others show a value for the molecular lowering that is two or three times the calculated value, while others still, such as aluminium chloride and nitrate, with as large amount of water of crystallisation as that possessed by any chloride or nitrate studied, and which are quaternary electrolytes, show even greater lowering as compared with the calculated value. These and many other relations are fully developed in our next paper to which reference has already been made.

We think we have experimental evidence for the existence of hydrates in concentrated solutions of nearly all the substances that we have studied. This is the first time that direct evidence for the existence of hydrates, in general, in concentrated aqueous solutions has been furnished.

The importance of this fact for physical chemistry is very great indeed. It has been said, and justly said, that the relations between gases and solutions, of which the physical chemists make so much, apparently hold only for very dilute solutions. The reason why these relations apparently do not hold in concentrated solutions is that, in such solutions, the dissolved substance combines with a part of the solvent, forming hydrates with it, and thus diminishes the amount of solvent present, acting as such.

When these hydrates are taken into account it is more than likely that the gas laws will be shown to hold for concentrated solution as well as for dilute; especially if the gas laws are applied to concentrated solutions as to concentrated gases, in accordance with the equation of Van der Waals.

Having established, to our own satisfaction at least, the existence of hydrates in concentrated solutions of electrolytes, we calculated the composition of such hydrates in a number of cases. We find that the most concentrated solution contains the most complex hydrate, just as we would expect, since dilute solutions cannot contain hydrates of any appreciable complexity, as is proved by the fact that the freezing-point method and the conductivity method of measuring dissociation give the same results. We have also calculated the composition of the hydrates formed by a given substance at various concentrations, and have plotted the results in a curve—one ordinate being the composition of the hydrate, and the other the concentration of the solution. To our delight every substance thus dealt with gave a perfectly smooth curve, notwithstanding the fact that every point on the curve contained four experimentally determined values.

These facts will all appear when our paper (*Am. Chem. Journ.*, April, 1904) describing this work in full is published in the near future.

Chemical Laboratory,  
Johns Hopkins University, Baltimore, U.S.A.  
January, 1904.

## CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS.\*

### A STUDY OF NEODYMIUM: PREPARATION OF PURE MATERIAL—EFFORTS TO DECOMPOSE IT INTO ITS CONSTITUENTS.

By CHAS. BASKERVILLE and RESTON STEVENSON.

**Synopsis.**—This paper presents a new and rapid method of freeing neodymium from lanthanum, viz., fractional precipitation of the chloride solution by saturation with gaseous hydrochloric acid. A number of fractionations of the purified material were made to prove the complexity of neodymium, but without success. The methods used were, fractional precipitation by gaseous hydrochloric acid (over 100); partial decomposition by fusion of the double alkaline nitrates (23); precipitation by primary ammonium oxalate (31); solution in ammonium carbonate and precipitation with acetic acid (3); fractional precipitation by such organic bases as aniline (4), benzylamine (29), piperidine (19), and phenylhydrazine (8).

Naturally investigations were to be carried along with the foregoing, looking toward the preparation of pure neodymium compounds. It was noted that the preparation of pure neodymium salts, according to Auer's method, presented numerous difficulties, and required much time and many repetitions of the process. Boudouard (*Comptes Rendus*, cxcvi., No. 12; *CHEMICAL NEWS*, lxxvii., 193) has shown that neodymium gives a double sulphate with potassium more soluble in water than that of praseodymium, and presents a more rapid separation than is perhaps possible by fractional crystallisation. Muthmann and Rölzig (*Ber. d. Chem. Ges.*, 1898, xxxi., 1718–1731), working with didymium, had from yttria, extracted from monazite sand, found, after making sixty crystallisations, a neodymium sulphate containing only 0.3 per cent of praseodymium. The oxide,  $\text{Nd}_2\text{O}_3$ , obtained by them was described as "fast weiss"—(see below).

Reference has been made to the tentative acceptance of neodymium as an element. The agreement of the figures given for the atomic weight of neodymium by Jones (*Am. Chem. Journ.*, xx., 345), Brauner (*Proc. Chem. Soc.*, 1898, No. 191, p. 70), and von Scheele (*Zeit. Anorg. Chem.*, xvii., 310) point clearly to a constant material obtained from different sources. The same was also true of didymium before the classical work of Auer von Welsbach (*loc. cit.*). Urbain (*Bull. Soc. Chim.*, [3], xx., 9; *CHEM. NEWS*, lxxviii., 74), in studying didymium, had from yttria extracted from monazite sand, found not only neodymium, but such other earths as erbium and Soret's X (holmium), members of the terbic group and a little cerium. While absorption band 469 was distinctly visible, 482 and 444 were doubtful (see papers following). Demarcay (*Comptes Rendus*, civ., 580) and Krüss and Nilson (*Ber. d. Chem. Ges.*, xx., 2124) had previously made analogous observations with variations of the relative intensities of these bands. Later, Demarcay (*Comptes Rendus*, cxcvi., 14; *CHEMICAL NEWS*, lxxvii., 219) showed that some absorption bands have been found in didymium which appear to belong neither to neodymium nor praseodymium. Further, the presence of a certain amount of samarium causes the blue bands to disappear in a vague, hardly visible nebulosity. He concluded, as a result of a number of fractionations by different methods and examinations of the absorption bands, spark spectra (including the ultra-violet region), fluorescent spectrum in calcium sulphate, and by spectral colorimeter, "in spite of statements of several chemists, neodymium is a simple body and not a mixture." Yet the work by Brauner (*Proc. Chem. Soc.*, March 31, 1901) on variations in the oxides gives evidence of the complexity of that element.

\* Presented in abstract at the Cleveland Meeting of the American Chemical Society. From the *Journal of the American Chemical Society*, 1904, xxvi., No. 1.

Our investigation was begun with the intention of testing the nature of neodymium. Many have difficulty in assigning neodymium a satisfactory location in the periodic system. Therefore, in the event we learned that neodymium was simple, it was highly desirable to determine positively its place in the natural system.

Obviously the first step was to prepare neodymium as pure as possible. The constant associates of neodymium are lanthanum and praseodymium, which are separated by methods which, at best, are long, slow, and difficult of execution. It is very desirable, therefore, to secure a method thorough and quick for the absolute elimination of these accompanying substances.

500 grms. of crystalline, double ammonium neodymium nitrate were used during the preliminary trials of different methods.\*

#### Partial Decomposition of Double Nitrates by Fusion.

The classic method of Berlin (*Scand. Naturf.*, 8 Möde Kjöbenhavn, 1860, p. 448) was applied to 200 grms. of the ruby-coloured crystals which had lanthanum as the main impurity and some praseodymium. The numerous variations of the directions given by Berlin, offered by subsequent workers, looking toward uniform heating and decomposition, failed to improve the process. At best, such methods of fractionation required a great amount of time and patience. The material was carried through twenty-three fractionations, and, while much of the lanthanum was removed, the neodymium still contained praseodymium.

Hood (*CHEMICAL NEWS*, lii., 271), discussing principles of fractioning, observed:—"It is evident then that fractional precipitation is a method for separating two oxides differing but slightly in basic properties. It may be the work of a lifetime, even when working on a large amount of material." Having the latter and not being assured of the temporal allotment of a chemist (thirty years), we looked for more encouraging methods.

#### Saturation of Neodymium Chloride Solution with Hydrochloric Acid.

The neodymium ammonium nitrate containing lanthanum as chief impurity with some praseodymium was converted into the chloride by the precipitation as hydroxide with ammonia, and washing free of the precipitant. It is inadmissible to convert the double salt to the oxide by direct ignition in platinum, as the metal is attacked. To be sure, it would appear that this contamination could be readily removed by subsequent solution of the oxide in hydrochloric acid. In practice, however, the platinum is found in the solution. The explanation of this is easily had. In the first place, Brauner (*Proc. Chem. Soc.*, March, 1901; *CHEMICAL NEWS*, lxxxiii., 197) has shown the formation of a dioxide when the nitrate is ignited. This oxide causes the evolution of chlorine on dissolving it in hydrochloric acid with the consequent solution of the platinum. In the second place, the metal is finely divided, and W. L. Dudley (*Journ. Am. Chem. Soc.*, 1893, xv., 272) and Mallet (*Am. Chem. Journ.*, 1901, xxv., 430) have shown that platinum in this condition and exposed to the air is soluble in hydrochloric acid. In later work, where large quantities were used, large earthenware tubs of 100 litre capacity with a spiral row of decantation openings on one side were used. The hydroxide was allowed to settle and supernatant liquid drawn off, and the hydroxide washed twice with water by decantation. The decantations were filtered free of any suspended hydroxide, and the filtrate discarded.

It may be well here to call the attention of those beginning work on the rare earths to the advisability of pursuing the very safe plan of saving all filtrates until they are thoroughly tested. These filtrates were not thrown away unless 10 to 20 litres had been evaporated and ignited,

and showed only a trace of residue. In many cases the clear water became turbid on standing exposed to the air. This was no doubt due to the absorption of carbon dioxide, which caused the precipitation of the neodymium compound or solution of the hydroxide in the wash-water and subsequent precipitation on neutralisation by the excess of ammonium hydroxide in the filtrate. The hydroxide was thrown upon a filter, dried, and ignited. The unglazed porcelain filter was eventually utilised in this work, as with the praseodymium, giving most satisfactory results.

The dried hydroxide was ignited in platinum over powerful Bunsen burners until no more fumes arose. The ignited product was pulverised in an agate mortar, and added to the concentrated hydrochloric acid in large porcelain casseroles. The solution, which was facilitated by heat, exhibited dichroism, presenting the colour of juniper swamp water (yellowish brown) with transmitted light, and purple with reflected light (especially noticeable upon pouring), and faintly purple on dilution.

The neodymium chloride was filtered to remove a small grey residue of platinum obtained from the vessels, and concentrated until a crust of crystals formed on top of the solution. On cooling, yellow-brown crystals appeared abundantly. They were separated, dissolved in water, and the mother-liquor of neodymium chloride separately fractionated by saturation with hydrochloric acid gas.

This method of treatment possesses nothing essentially new, having been fully described previously by Dennis (*Journ. Am. Chem. Soc.*, 1902, xxiv., 421, and previously). The gas was generated by allowing concentrated hydrochloric acid to fall dropwise upon concentrated sulphuric acid. The generator was provided with a safety-flask, and the gas let into the solution by means of an inverted funnel or thistle tube, which just dipped underneath the surface of liquid.

During the early work and with the small quantities of material, only pure acids were used. When, however, later, large quantities of material (several carboys of acids) were employed, on account of the expense, commercial reagents were had recourse to. A satisfactory generator of gaseous hydrochloric acid from commercial acids was devised by one of us (Stevenson), and W. M. Marriott (of this laboratory), and is described elsewhere.

The solution was saturated with the gas until crystals appeared. The supernatant liquid was carefully decanted, the crystals washed with concentrated chemically pure hydrochloric acid, and the liquid again decanted from any crystals which subsequently formed in the mother-liquor. It was later learned that by allowing the solution to cool, the two crops of crystals could be collected in the one beaker, and washed several times with hydrochloric acid; the mother-liquor was subjected to another fractionation, crystals re-dissolved in the least amount of water, and fractionated again. Whenever solutions became too weak to be further fractionated, they were concentrated, and subjected to the action of the gas.

During the earlier part of the work, forty-five fractionations were made, and the resulting fractions classified according to the absorption spectra into four large solutions; the extreme crystals, medium crystals, medium solutions, and extreme solutions, and into two small solutions—the pene-extreme crystals and the pene-extreme solutions. These solutions were fractionated as before, except that the application of the gas was continued until no more crystals appeared, and the "criss-crossing" preceded each fractionation.

After twenty-two more of such fractionations, there resulted four fractions:—Extreme crystals, a very large fraction; medium crystals, about two-thirds as large; medium solutions, a moderate-sized fraction; and extreme solutions, small fraction. The extreme crystals in neutral nitrate solution showed an absorption spectrum identical with the spectrum of the extreme solution, except that in the latter the band  $\lambda$  460–466 had disappeared. This band belongs to samarium. The arc spectrum of the oxide from the extreme crystals showed only vanishing traces of

\* Obtained through the generosity of Dr. H. S. Miner, chemist of the Weibach Lighting Co.

lanthanum.\* The oxide of the extreme solution was brown and violet. The brown portion was largely lanthanum and neodymium with some praseodymium. The method therefore was promising, as almost pure neodymium was obtained in the heads.

It may be well to state the result of some of our observations here for the guidance of others in the application of this method. The method was improved as follows:—The concentrated neodymium chloride solution was saturated with hydrochloric acid gas until no more crystals appeared. These crystals were then dissolved to a saturated aqueous solution, and refractioned. After seven such complete precipitations, the last crystals were absolutely free from lanthanum, according to the arc spectrum. This method was used on a large scale subsequently, working with as much as 8 kilograms. of neodymium ammonium nitrate, using the hydrochloric acid generator of Stevenson and Marriott. Within a week, a kilogram. of neodymium oxide absolutely free from lanthanum was obtained. This serves to illustrate the value of the method.

#### Remarks.

During all our investigations with the rare earths, on account of their value, the fragile containers are always placed in a second large vessel in case of accidental breakage.

The precipitation with the hydrochloric acid gas sometimes began immediately, generally in about twenty minutes. In a few cases, where the solution was dilute, only after two hours, and in a few very dilute solutions not at all. The mother-liquor decanted from crystals frequently yielded another crop on standing. Oftentimes, apparently, a supersaturated solution would be had which, when poured from one vessel to another, gave rise to spontaneous crystallisation.

The precipitates were always crystalline. Rapid precipitation from saturated solutions gave trellised translucent crystals which appeared white in the mother-liquor. Vitreous needles resulted from the concentrated solutions; fine iridescent crystals from dilute solutions. The spontaneous crystals, especially when formed after days of standing, were handsome pyramids from 4 to 7 m.m. in length. The crystals were always more or less coloured; the extreme crystals were redder, and the crystals from the extreme solutions were yellowish. Sometimes the crystals appeared in alternate layers of rose and white, very likely due to a different manner of precipitation, because no difference was observed in the absorption spectra of solutions made from the two. This is no proof, however, that different substances did not separate out more or less contaminated with each other, for, as is well known, the absorption spectrum is not a final test of a fractionation. These observations were made near the time when it would become necessary for one of the authors (S.) to leave this laboratory, so that checks could not be made by means of atomic weight determinations. This work will be taken up later.

The colours of the solutions obtained in the fractionations were striking. The characteristic dichroism of the original chloride solution has been adverted to. The extreme solutions were a bright yellow in transmitted light, and bright green in reflected light. The criss-crossing was determined by the colour of the solutions; and the classification by colour, location in the chart of the fractionations, but mainly by the absorption spectra. All these methods agreed.

The absorption spectra at first were taken of fractions as obtained without any attempt at uniform concentration. As a result, the spectra were deceptive, and not comparable. Subsequently a basis was assumed for all comparisons, 1 grm. of oxide being converted into neutral chloride and diluted to 10 c.c. An interesting observation was made in

the examination of the fractions by means of the absorption spectrum, for band  $\lambda$  460—466, the strong band belonging to samarium, according to Thalén, disappeared coincident with the loss of the yellow purple dichroism.

The bands  $\lambda$  569, 482, 469, and 443, characteristic of praseodymium, persisted throughout; yet, according to the arc spectrum, the praseodymium was present in a very small amount—less than 1 per cent as shown by Jones (*Am. Chem. Journ.*, xx., 345). We were so fortunate as to have in our possession some of the material used by Dr. H. C. Jones in his careful atomic weight determinations of neodymium and praseodymium. The arc spectrum of the purest preparation showed traces of gadolinium and samarium in both samples, but by a comparative examination of a solution of samarium, the absorption lines of this element were not found as impurities in our purest neodymium.

The colour of the oxides obtained from the different fractions presented an interesting study. Demarçay (*Comptes Rendus*, cxxvi., 14; *CHEMICAL NEWS*, lxxvii., 219) states that pure neodymium is a violet-coloured oxide. A pale violet oxide was given by the extreme crystals, but a lump of it had a brown core, which easily became violet upon ignition (compare Muthmann and Rölig above). This same oxide upon recovery after use for precipitation by organic bases (see below) was a mixture of brown cinders, rose-coloured scales, mole-skin dust, and black specks; yet each of these, carefully picked out and separately examined, gave the same absorption bands—the true spectrum of our purest neodymium. The oxides of the extreme solutions was a mixture of lavender and brown. It sometimes became almost white upon vigorous ignition, indicating the predominance of lanthanum. Certain blue oxides were also obtained, and when sufficient shall have accumulated, will be examined (see *Journ. Russ. Phys. Chem. Soc.*, xxix., 209). Perhaps this may prove to be Chroustachoff's glaukodymium. The dark oxides go into solution in nitric acid more energetically than the lavender-coloured oxides, a temporary rose-coloured residue being obtained.

#### Further Experiments looking to Proof of Complexity of Neodymium.

Having secured pure neodymium containing only a trace of praseodymium, we applied a few methods looking toward a determination of the nature of that element. Below are given the methods used.

#### Precipitation by Primary Ammonium Oxalate.

A neutral neodymium chloride solution prepared from the pure oxide obtained according to the method given above was used. A saturated aqueous solution of ammonium oxalate was added until a permanent cloudiness was obtained. About 5 c.c. of saturated primary ammonium oxalate was then added, giving local turbidity, which became a mealy precipitate upon stirring. This was essentially the method used by Dennis and Dales in their work upon the purification of yttrium (*Journ. Am. Chem. Soc.*, 1902, xxiv., 425). After twelve hours, the precipitate settled as a rose-coloured powder, leaving the supernatant liquid clear. Thirty-one such fractionations were made. The last two fractions were obtained as follows:—To the boiling mother-liquor 10 c.c. of the primary oxalate were added, and no precipitate formed, but on cooling, crystals gradually appeared. The plan of adding praseodymium oxalate crystals to induce a separation of that constituent of didymium was tried, but the solution was exhausted. The final mother-liquor was evaporated to dryness, ignited, converted into neutral chloride solution, and examined with the spectroscope. All of the mother-liquors obtained throughout the fractioning were similarly treated, as well as the precipitates. The same absorption spectrum was obtained in every case; therefore, the process was not tried more elaborately.

\* These determinations were made by Dr. W. J. Humphreys, of the University of Virginia, with the same instrument as mentioned in the Praseodymium paper.

*Solution in Ammonium Carbonate and Precipitation by Acetic Acid.*

Pure neodymium hydroxide obtained from the chloride above by precipitation with ammonia, was washed twice by decantation. 400 c.c. of water saturated with pure ammonium carbonate were added. No perceptible solution of the hydroxide was observed. The liquid was poured off and the hydroxide washed three times by decantation. The liquid gave no precipitate with acetic acid, but on evaporation and ignition a very small residue was obtained. Converted into a neutral chloride solution, the liquid showed only the stronger absorption bands of neodymium. It was too small in amount to be made up to the standard strength adopted. The neodymium hydroxide was treated once more with 300 c.c. of saturated ammonium carbonate or several days with a similar result, consequently it was abandoned.

*Fractional Precipitation by Organic Bases.*

G. Krüss (*Zeit. Anorg. Chem.*, 1893, iii., 108) fractionated certain of the rare earths with such substituted ammonias as aniline. Smith and Jefferson (*Journ. Am. Chem. Soc.*, 1902, xxiv., 540) published the effect of precipitating the rare earths with aromatic bases. We endeavoured to utilise their observations in making fractional precipitations similar to the work of Schutzenberger and Boudouard, and other workers in fractioning the rare earths with ammonia. E. T. Allen later used certain weak organic bases for separating thorium and zirconium from iron and beryllium (*Ibid.*, 1903, xxv., 421).

**Aniline.**—To a moderately concentrated, slightly acid solution of neodymium chloride, aniline was added in excess, with stirring. No precipitate appeared at once. The solution was concentrated, and more aniline added. After three days, a slight white precipitate formed, adhering to the beaker. It was filtered, and the filtrate treated with aniline, and boiled. More aniline was added, and another meagre white precipitate formed. Further attempts to get a larger precipitate, even after allowing the solution to stand for three months, failed. These three precipitates showed similar absorption bands, and were identical with the spectrum of the diluted mother-liquor. The process was unpromising, and therefore discontinued.

**Benzylamine.**—Benzylamine upon addition to a neutral neodymium chloride solution immediately gave a rose-coloured, flocculent precipitate, the liquid becoming somewhat cloudy upon stirring. The precipitate settled after twelve hours, and was very difficult to filter. Only eight drops of the organic base were used to get a fractional precipitation, because, as stated by Smith and Jefferson, the precipitation is quantitative (*loc. cit.*) After twenty-nine fractionations, the solution was exhausted. Spectroscopic examination of the precipitates, the mother-liquors, and the final residue, according to the methods already cited, showed nothing noteworthy, and the process was not further elaborated.

**Piperidine.**—Piperidine acted similarly to benzylamine, nineteen fractions being carried out. The mother-liquor soon assumed a bright yellowish green colour. Some of the hydroxides were rose colour and some were almost pale yellow.

**Phenylhydrazine.**—The phenylhydrazine was added in an excess to a neutral chloride solution. Gradually, in the course of twenty-four hours, there formed a dark red precipitate which adhered to the beaker, and was difficult to filter. The precipitate was dissolved in concentrated nitric acid, and gave a dark red solution due to the action of the acid upon the organic base, or a decomposition product of the base. Seven fractionations, each one requiring a week, were carried out. The precipitates showed no divergence from the original, when in the form of neutral chloride solutions they were examined with the spectro-scope. After the third precipitation, the filtrate became so turbid, either from decomposition products of the phenylhydrazine, or through hydrolysis of such double compounds

as are mentioned by Delafontaine, that the process was given up.

*Summary.*

In endeavouring to prove the complexity of neodymium by fractional precipitation, in our hands the following methods failed:—Fusion of the double nitrate, according to Berlin (see preceding paper); precipitation by primary ammonium oxalate; solution in ammonium carbonate and precipitation by acetic acid; fractional precipitation by gaseous hydrochloric acid; precipitation by the organic bases—aniline, benzylamine, piperidine, and phenylhydrazine.

The following methods are among those which will be tried in this laboratory:—The use of sodium acetate and hydrogen peroxide, electrolysis, reduction by metallic magnesium, magnesium usta, treatment with mercury oxide and nitrate, copper oxide (method applied by Schutzenberger and Boudouard to cerium), dialysis, and ammonium persulphate.

NEW FORMS OF PIPETTES.

By B. M. MUKERJEE.

I HAVE been using several forms of pipettes for drawing bromine, chlorine-water, &c., which I have not seen described anywhere. The following descriptions may be useful.

The pipette, Fig. 1, consists of a reservoir, A, containing water; a tube, B, with a bulb, b, of about the same capacity

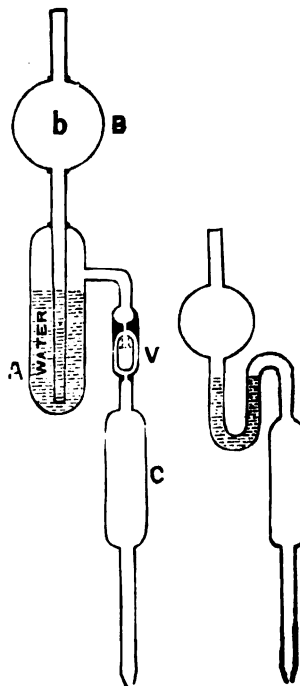


FIG. 1.

FIG. 2.

as the reservoir, passing nearly to the bottom of the reservoir; and a pipette, C. For greater safety a valve, V, is attached to the pipette, as shown in Fig. 1; it is, however, often omitted. On drawing the water into the bulb, b, the liquid rises in the pipette, C, without the fumes coming in contact with the mouth.

A simpler form of the above is shown in Fig. 2, where the U-tube serves the purpose of the reservoir in Fig. 1. This form is not quite so convenient as the other.

Thomason College, Roorkee.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.

By CHARLES BASKERVILLE, Ph.D., F.C.S., Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 151).

## LIST OF PROPOSED CHEMICAL ELEMENTS (continued).

| Date. | Name.           | Source.                                | Authority.            | Reference.                                                                                                                         | Remarks.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
|-------|-----------------|----------------------------------------|-----------------------|------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1842  | Didymium.       | Lanthana.                              | Mosander.             | Liebig's Ann. Chem., 44, 125; and 48, 210.                                                                                         | Doubted by Hermann (Journ. Prakt. Chem., 1845, 34, 182). Shown to contain samarium; later split into neo- and praseodymium by Auer (Monats., 6, 477); Delafontaine had written on the probable compound nature (Comptes Rendus, 1878, 87, 634). Krüss and Nilson, from study of absorption spectrum, maintained composed of nine elements (Chemical News, 1887, 56, 166). See papers by Baskerville for summary of spirited controversy by numerous workers as to this. Mosander separated yttria into basic yttria, least basic erbia, and intermediate terbia. Shown by Berlin (1860) to have no existence, although Mosander's results were confirmed by Berzelius ("Lehrbuch," 2nd French edition, 2, 163). Svanberg and Scheerer, Berlin did obtain rose-coloured salts of terbia, since called erbium, but Mosander's yellow erbia, the present terbia, he did not secure (Scand. Naturf. 8 mode Kjöbenhavn, 1860, p. 448). Verified by Delafontaine, Bahr and Bunsen (Liebig's Ann., 1866, 137), and Cleve and Högblund (Bull. Soc. Chim., 1872, 121, 18, 193, 289). |
| 1843  | TERBIUM.        | With erbium in gadolinite. Gadolinite. | Mosander.             | Ann. Chem. Pharm., 68, 131, 137, &c.                                                                                               | Reason for assuming existence, "great variation in atomic weight of oxide." Denied by Berlin (Journ. Prakt. Chem., 58, 145); also Marignac (Comptes Rendus, 50, 952; Chem. Centrbl., 1866, 603). Proved identical with zirconium by Hermann (Journ. Prakt. Chem., 97, 330; Jahrsb., 1866, 189). Taken back by Svanberg, (Journ. Prakt. Chem., 58, 145).                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |
| 1843  | ERBIUM.         |                                        | Mosander.             | Ann. Chem. Pharm., 68, 131, 137, &c.                                                                                               | Discoverer later showed pelopid acid was convertible into columbic and hypocolumbic acids, hence non-existent.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
| 1845  | NORIUM.         | Zirconia.                              | Svanberg.             | Oefvers. K. Vet. Akad. Forh., 1845, 34; Pogg. Ann., 65, 317; Am. Journ. Sci., [2], 1, 257; Berg. Jahrsb., Prakt. Chem., 57 and 97. | Proved to be columbium.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |
| 1846  | Pelopium.       | Bavarian tantalate.                    | H. Rose.              | Pogg. Ann., 69 and 90; Chem. Gaz., 1846, Sept. 5; Am. Journ. Sci., [2], 3, 357.                                                    | Proved to be composed of iron, chromium, and phosphoric oxides (1854).                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 1846  | Ilmenium.       | Ilmenite.                              | Hermann.              | Journ. Prakt. Chem., 38 and 40; Pogg. Ann., 73.                                                                                    | Proved to be thorium (1863).                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |
| 1850  | Aridium.        |                                        | Ullgren.              | Journ. Prakt. Chem., 52; Ann. Chem. Pharm., 76 and 78.                                                                             |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| 1851  | Donarium.       |                                        | Bergmann.             | Ann. Ch. Pharm., 80 and 84.                                                                                                        |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| 1852  | Thalium.        |                                        | Owen.                 | Am. J. Sci., [2], 13, 16, 17.                                                                                                      | Two grains white metal like platinum from California gold, with properties unlike those of any known element.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |
| 1852  | Unnamed.        | Gold ores (Calif.).                    | Genth.                | Proc. Acad. Sci. Phil., 9, 209; Am. Journ. Sci., 1853, [2], 15, 446.                                                               | Precipitation by potassium ferrocyanide. Specific gravity oxide 5.5 instead of 4.3.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| 1854  | Nameless earth. | Zirconia.                              | Sjogren.              | Journ. Prakt. Chem., 55 and 57.                                                                                                    | Setterberg obtained metallic caesium by electrolysis in 1881.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |
| 1860  | Caesium.        | Alkaline salts.                        | Bunsen and Kirchhoff. | Pogg. Ann., 113, 337; Chem. News, 11, 281.                                                                                         |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |

|             |                           |                                   |                           |                                                                                                                                      |
|-------------|---------------------------|-----------------------------------|---------------------------|--------------------------------------------------------------------------------------------------------------------------------------|
| 1860        | Dianium.                  | Finnish tantalate.                | Von Kobell.               | Ann. Chem. Pharm., 114 and 136; Journ. Prakt. Chem., 83, 193, 449.                                                                   |
| 1861        | RUBIDIUM.                 | Lepidolite.                       | Bunsen and Kirchhoff.     | Jahrb., 173; Chemical News, 3, 357.                                                                                                  |
| 1861        | Nameless earth. THALLIUM. | Calcium group. Selenium residues. | Dupré. Crookes.           | Chemical News, 3, 193, 303.                                                                                                          |
| 1862        | Unnamed.                  | Native platinum (Oregon). Wasite. | C. F. Chandler.           | Am. Journ. Sci., [2], 32, 351.                                                                                                       |
| 1862        | Wastium.                  |                                   | Bahr.                     | Pogg. Ann., 119, 572; Journ. Prakt. Chem., 91, 316.                                                                                  |
| 1863        | INDIUM.                   | Lead, zinc, and arsenic ores.     | Reich and Richter.        | Journ. Prakt. Chem., 89, 441.                                                                                                        |
| 1864        | A new earth.              | Calcareous mineral.               | Bischoff.                 | Pogg. Ann., 122, 646; Am. Journ. Sci., 1865, [2], 38, 420.                                                                           |
| 1864        | Nameless earth. Nigrium.  | Zirconia.                         | Nylander.                 | Acta. Univers. Lundensi, 1864.                                                                                                       |
| 1866        | Aurorium.                 | Zirconia.                         | Church.                   | Chemical News, 1866.                                                                                                                 |
| 1867 (1898) | Nebulium.                 | Aurora.                           | Angström.                 | Nov. Acta. Upsala Sci., [3], 9, 29.                                                                                                  |
| 1868 (1895) | HELIUM.                   | Sun's chromosphere, Cleveite.     | Janssen, Lockyer, Ramsay. | "Gases of the Atmosphere," Ramsay.                                                                                                   |
| 1869        | Yargonium.                | Zirconia.                         | Sorby.                    | Proc. R. S., 17, 511; Chem. News, 19, 121, 142, 181; Ber., 1, 126, 193, 337, 382.                                                    |
| 1869        | Nameless earth.           | Zirconia.                         | Loew.                     | Annals N.Y. Lyc. Nat. Hist., 9, 211.                                                                                                 |
| 1875        | GALLIUM.                  | Gallium.                          | De Boisbaudran.           | Comptes Rendus, 81, 493; Chem. News, 32, 159.                                                                                        |
| 1877        | Davyum.                   | Platinum residues.                | Kern.                     | Chem. News, 36, 4, 92, 155, 164; and 37, 65.                                                                                         |
| 1877        | Neptunium.                | Columbite and ferromenite.        | Hermann.                  | Journ. Prakt. Chem., [2], 15, 105; Chem. News, 35, 197.                                                                              |
| 1877        | Mosandrium.               | Samaraskite.                      | J. L. Smith.              | Letter No. 14 in Am. Journ. Sci., [3], 14, 509; Proc. Acad. Nat. Sci. Phila., 29, 194; Comptes Rendus, 87, 148; Chem. News, 38, 100. |

(To be continued).

Identity questioned by H. Rose, Ste.-Claire Deville, and Hermann. Gives deep blue colour with tin and hydrochloric acid. Marignac and Blomstrand proved it to be columbium (1865).  
Beketoff prepared rubidium by heating hydroxide with aluminium (1888).  
Brilliant green line spectrum. Lamy announced same element about same time (Ann. Chem. Phys., [3], 67, 385). Probably same as that discovered by Gentz, as pointed out by Chandler.  
Nickles showed it to contain yttrium, terbium, and didymium (Comptes Rendus, 57, 1740). Delafontaine maintained it to be cerium oxide, while Popp contended it was a mixture of oxides of yttrium, cerium, and didymium (Ann. der Chem. und Pharm., 141, 364, 568). Blue line (hence name) found in spectrum when looking for thallium.  
Forms characteristic volatile chloride, which is soluble in water precipitated by bases. This dissolves in hydrochloric acid, forming the chloride, which sublimes easily.  
Not verified.  
Solubility of double potassium sulphates.  
Black absorption bands of spectrum.  
"Still awaiting discovery by more fortunate spectroscopists are the unknown celestial elements, aurorium, with a characteristic line at 5507.7; and nebulium, having two bright lines at 5007.05 and 4955.03."—Crookes, 1898.  
See Huggins (Roy. Soc., 1899; Chemical News, 59, 101), with account of observations in 1874.  
D<sub>3</sub> line characteristic in this celestial and terrestrial element, doubtless had by Palmieri (1882) from lava, and Hillebrand from uranium minerals. Ramsay secured it and described its properties definitely, however.  
Absorption bands in spectrum. Solubility chloride in hydrochloric acid.  
Variation in nature of sulphates. Solubility oxide in oxalic acid.  
Found by spectroscopy; in very small amounts, but widespread, as shown by Hartley and Ramage. Quite true of most elements (see Gautier on hydrogen and arsenic; Baskerville on Titanium).  
Shown by Mallet to be a mixture of iridium, rhodium, and iron (Am. Chem. Journ., 1898, 20, 776).  
Separated by difference in solubility of double potassium fluorides.  
Due to insolubility of double potassium sulphate. Marignac proved it identical with terbium (Compt. Rend., 87, 281). Also by Delafontaine (C. R., 87, 600). De Boisbaudran finally demonstrated it to be mixture of didymium, samarium, gadolinium, and terbium (loc. cit., 102, 647).



ELEVENTH ANNUAL REPORT OF  
THE COMMITTEE ON ATOMIC WEIGHTS.  
DETERMINATIONS PUBLISHED DURING 1903.\*

By F. W. CLARKE.

DURING the year 1903 comparatively few determinations of atomic weight have been published, but some of the work done is of notable importance. Other work is known to be in progress, and the output for the coming year should be of considerable magnitude. The results which have appeared so far are summarised below.

*Potassium and Cesium.*

The memoir by Richards and Archibald (*Proc. Amer. Acad.*, xxxviii., 443; also *Zeit. Anorg. Chem.*, xxxiv., 353) relates primarily to cesium, but some determinations in regard to potassium were made incidentally. First, potassium chloride, dried at 700°, was precipitated by silver nitrate. The latter had been prepared from a known quantity of pure silver, so that two distinct ratios were determined. Vacuum weights are given throughout, and the reductions were made with  $\text{Ag} = 107.930$ , and  $\text{Cl} = 35.455$ ;  $\text{O} = 16$ . For the ratio between  $\text{KCl}$  and  $\text{AgCl}$  we have—

| Weight $\text{KCl}$ . | Weight $\text{AgCl}$ . | Atomic weight $\text{K}$ . |
|-----------------------|------------------------|----------------------------|
| 2.50019               | 4.80600                | 39.137                     |
| 2.50391               | 4.81325                | 39.136                     |

For the ratio  $\text{Ag} : \text{KCl}$  the data are—

| Weight $\text{KCl}$ . | Weight $\text{Ag}$ . | Atomic weight $\text{K}$ . |
|-----------------------|----------------------|----------------------------|
| 2.50019               | 3.61747              | 39.140                     |
| 2.50391               | 3.62283              | 39.141                     |

Mean of both series,  $\text{K} = 39.139$ .

Another set of measurements was made by an entirely new method. Potassium nitrate was ignited with weighed quantities of silica;  $\text{K}_2\text{O}$  as silicate remained, and  $\text{N}_2\text{O}_5$  (or, rather, its decomposition products) was driven off. From the ratio thus ascertained,  $\text{K}_2\text{O} : \text{N}_2\text{O}_5$ , the atomic weight of potassium was calculated with  $\text{O} = 16$  and  $\text{N} = 14.040$ . The data are as follows:—

| Weight $\text{KNO}_3$ . | $\text{N}_2\text{O}_5$ lost. | Atomic weight $\text{K}$ . |
|-------------------------|------------------------------|----------------------------|
| 1.81034                 | 0.96692                      | 39.138                     |
| 3.14564                 | 1.68005                      | 39.142                     |
| 2.55598                 | 1.36512                      | 39.142                     |
| Mean .. ..              |                              | 39.141                     |

For the work on cesium the material was purified by repeated re-crystallisation of the dichloride,  $\text{CsCl}_2\text{I}$ . By heating this salt to from 90° to 100° in an electric oven it was reduced to  $\text{CsCl}$ , which was spectroscopically free from other alkaline salts. Several samples of chloride were prepared, and several series of determinations were made, similar to the two sets of chloride analyses given under potassium. The data thus obtained are as follows:—

| Ratio $\text{AgCl} : \text{CsCl}$ . |                        |                             |
|-------------------------------------|------------------------|-----------------------------|
| Weight $\text{CsCl}$ .              | Weight $\text{AgCl}$ . | Atomic weight $\text{Cs}$ . |
| <i>First Series.</i>                |                        |                             |
| 3.83054                             | 3.26240                | 132.901                     |
| 3.95120                             | 3.36532                | 132.892                     |
| 4.27237                             | 1.93555                | 132.882                     |
| 3.02935                             | 2.58003                | 132.901                     |
| 3.19774                             | 2.72382                | 132.878                     |
| Mean .. ..                          |                        | 132.891                     |
| <i>Second Series.</i>               |                        |                             |
| 2.35068                             | 2.00253                | 132.858                     |
| 2.06245                             | 1.75678                | 132.878                     |
| 2.56372                             | 2.18358                | 132.892                     |
| Mean .. ..                          |                        | 132.876                     |

*Third Series.*

|         |         |         |
|---------|---------|---------|
| 2.01881 | 1.71972 | 132.868 |
| 1.77391 | 1.51093 | 132.886 |

Mean .. .. 132.877

*Fourth Series.*

|         |         |         |
|---------|---------|---------|
| 3.08160 | 2.62484 | 132.881 |
| 3.13117 | 2.66720 | 132.872 |
| 5.06656 | 4.31570 | 132.876 |

Mean .. .. 132.876

*Ratio  $\text{Ag} : \text{CsCl}$ .*

| Weight $\text{CsCl}$ . | Weight $\text{Ag}$ . | Atomic weight $\text{Cs}$ . |
|------------------------|----------------------|-----------------------------|
|------------------------|----------------------|-----------------------------|

*First Series.*

|         |         |         |
|---------|---------|---------|
| 3.83054 | 2.45600 | 132.880 |
| 3.95120 | 2.53351 | 132.871 |
| 2.27237 | 1.45686 | 132.891 |
| 3.02935 | 1.94244 | 132.868 |
| 3.19774 | 2.05023 | 132.883 |

Mean .. .. 132.878

*Second Series.*

|         |         |         |
|---------|---------|---------|
| 2.35068 | 1.50720 | 132.876 |
| 2.06245 | 1.32251 | 132.862 |

Mean .. .. 132.869

*Third Series.*

|         |         |         |
|---------|---------|---------|
| 2.01881 | 1.29434 | 132.886 |
| 1.77391 | 1.13743 | 132.871 |

Mean .. .. 132.878

*Fourth Series.*

|         |         |         |
|---------|---------|---------|
| 3.08160 | 1.97590 | 132.872 |
| 3.13117 | 2.00760 | 132.879 |
| 5.06656 | 3.24850 | 132.879 |

Mean .. .. 132.877

The general mean of these eight means gives  $\text{Cs} = 132.878$ .

By the ignition of calcium nitrate with silica the following data were obtained:—

| Weight $\text{CaNO}_3$ . | $\text{N}_2\text{O}_5$ lost. | Atomic weight $\text{Cs}$ . |
|--------------------------|------------------------------|-----------------------------|
| 3.76112                  | 1.04273                      | 132.882                     |
| 5.33334                  | 0.92416                      | 132.876                     |
| 4.81867                  | 1.33590                      | 132.886                     |
| 5.04807                  | 1.39960                      | 132.871                     |

Mean .. .. 132.879

Cesium bromide was also studied by Richards and Archibald, and two ratios are given by them. First, for the ratio  $\text{CsBr} : \text{AgBr}$ .  $\text{Br} = 79.955$ .

| Weight $\text{CsBr}$ . | Weight $\text{AgBr}$ . | Atomic weight $\text{Cs}$ . |
|------------------------|------------------------|-----------------------------|
| 3.49820                | 3.08815                | 132.878                     |
| 6.20409                | 5.47673                | 132.883                     |
| 7.17300                | 6.33213                | 132.880                     |

Mean .. .. 132.880

Secondly, for the ratio  $\text{CsBr} : \text{Ag}$ .

| Weight $\text{CsBr}$ . | Weight $\text{Ag}$ . | Atomic weight $\text{Cs}$ . |
|------------------------|----------------------|-----------------------------|
| 3.49820                | 1.77402              | 132.873                     |
| 6.20409                | 3.14606              | 132.885                     |
| 7.17300                | 3.63740              | 132.884                     |

Mean .. .. 132.881

The final values deduced by the authors from all their data are, with  $\text{O} = 16$ ,  $\text{K} = 39.140$ ,  $\text{Cs} = 132.879$ .

(To be continued).

\* From the *Journal of the American Chemical Society*, xxvi., No. 3.

# THE SPECIFIC HEATS OF METALS AND THE RELATION OF SPECIFIC HEAT TO ATOMIC WEIGHT.\*

## PART III.

By W. A. TILDEN, D.Sc., F.R.S.,  
Professor of Chemistry in the Royal College of Science, London.

THE object of the experiments of which an account is given in this paper was to determine whether the atomic heats of the elements entering into combination are preserved in the compound at all temperatures, previous results obtained by the author and others having shown that the specific heat of metals of small atomic weight, such as aluminium, increase very rapidly with rise of temperature.

As it is not possible to determine the specific heat of sulphur throughout a long range of temperature, tellurium was chosen for experiment. Compounds of tin, silver, and nickel with tellurium were prepared, and two alloys of silver and aluminium. The specific heats of all these elements were determined at intervals from the boiling-point of liquid oxygen to nearly 500° C. in the case of the less fusible elements, a range of about 680° C. From the mean specific heats the real specific heats at intervals of 100° absolute temperature were calculated, and from the specific heats the atomic heats were deduced. The mean specific heats of the compounds, formed by their union, were also determined, and from these data the molecular heats of the compounds calculated. On comparing the sum of the atomic heats of the elements present with the molecular heat of the compound at the successive temperatures, it was found that there is throughout a very close concordance. The order of difference may be shown by one example:—

### Nickel Telluride, NiTe.

| Temperature,<br>absolute. | Sum of atomic heat<br>of Ni and Te. | Molecular heat<br>of NiTe. |
|---------------------------|-------------------------------------|----------------------------|
| 100° .. .. .              | 9·20                                | 8·38                       |
| 200° .. .. .              | 11·08                               | 11·35                      |
| 300° .. .. .              | 12·22                               | 12·41                      |
| 400° .. .. .              | 13·00                               | 12·92                      |
| 500° .. .. .              | 13·49                               | 13·15                      |
| 600° .. .. .              | 13·85                               | 13·28                      |
| 700° .. .. .              | 14·11                               | 13·35                      |

The results of these experiments show that Neumann's law is approximately true, not only at temperatures from 0° to 100°, but at all temperatures. They also support the view that the specific heat of a solid is determined by the nature of the atoms composing the physical molecules, and is not a measure of the work done in expansion.

The paper concludes with a discussion of the relations of specific heat to atomic weight under different physical conditions—that is, in the solid, liquid, and gaseous states.

potassium thiocyanate, and ammonium thiocyanate were all tried, and the results compared. The first named was useless; of the others, which are all accurate, the thiocyanates gave the best results, and the ammonium salt was better than the potassium. With currents of 0·2 ampère per square decimetre the deposition of 0·05 to 0·08 grm. of gold was complete in five or six hours. With a current of 0·4 to 0·5 ampère, one and a-half to two hours sufficed. The presence of a little persulphate considerably reduced the voltage required. Experiments were also made to determine the best method of removing the deposited gold. Chlorine or bromine water were satisfactory, but slow; aqua regia was risky; the authors recommended a 2 per cent solution of potassium cyanide containing a little hydrogen peroxide, or a persulphate. One or two minutes then sufficed to remove the gold.

An improved form of basin electrode for working with hot solutions was described. A lamp-wick syphon from a small reservoir supplies water to compensate for evaporation.

Mr. CHAS. R. DARLING then read a paper on "*Thin-film Electrolysis, and a Proposed Application to Printing*," which was illustrated by experiments.

While investigating a process for letter-press printing by electrolysis without the use of ink—an extension of Bain's well-known telegraphic printing—the author found that the final results of electrolysis, when the electrolyte forms only a thin film, often differ materially from those observed in an ordinary cell. In these experiments a carbon or metal plate (it was immaterial which) formed the anode; on this was placed an impression pad, consisting of some sheets of moist blotting-paper; upon this was the trial sheet carrying the electrolyte film, and on this the cathode type or coin. Voltages from 6 to 200 were employed. To obtain a clear image of the type a certain minimum strength of solution is requisite. The first experiments were made with saline solutions; silver nitrate gave a clear permanent black image of the type, but the paper of course darkens on exposure; copper sulphate and nitrate yielded images that faded after a time; the same unexpected result occurred with lead, mercury salts, and bismuth. The best images were obtained with manganese salts. These, consisting as they did of the oxides or hydrates, were quite permanent; all purely metallic deposits, excepting silver, disappeared after a time. In the case of non-saline solutions the paper, which might consist of asbestos or pure Swedish filter-paper soaked in distilled water, acquired the properties of an exposed photographic plate, and on treating with a silver salt and developer, a perfect image of the cathode was obtained, even after a long interval. Reproductions of such "electrographs" are given in the paper. The latent images are not due to hydrogen peroxide, nor to metallic compounds, as they occur with carbon electrodes; but they reside in the surface of the paper in contact with or towards the cathode. The author ascribes them to a class of phenomena that have been investigated by Bose, and termed "the response of inert matter to electrical stimuli," and thinks they are probably the result of some state of strain set up in the film by the current. As regards the fading of the metallic images, this may be simply due to re-combination, although that explanation is not entirely satisfactory.

Dr. H. BORNES asked whether the author had treated his latent images with any solutions before developing.

Mr. N. J. BLUMAN said that in some electrolytic printing that he had carried out they had used some organic solutions with good results.

Mr. FONTANA suggested that the latent images might be due to occluded hydrogen.

Dr. PERKIN thought this supposition could be tested by exposing the paper to an oxidising agent.

Mr. BAWTREE asked whether it were possible to get rid of the latent images. If so, some light would be thrown on their cause.

Mr. PRIDEAUX asked whether the author had tried developing anode images where carbon formed the anode, so that no metal went into solution.

## PROCEEDINGS OF SOCIETIES.

### THE FARADAY SOCIETY.

Ordinary Meeting, March 21st, 1904.

The meeting was held at the Institution of Electrical Engineers, Dr. O. J. STEINHART in the Chair.

A paper by Dr. F. M. PERKIN and Mr. W. C. PREBBLE on "*The Electrolytic Analysis of Gold*" was read in abstract by Dr. PERKIN.

The object of the researches described was to arrive at an electrolytic method of estimating gold which should be perfectly accurate and yet far more rapid than the ordinary double cyanide method which the authors, differing from Claisen, consider inordinately long, even in hot solutions. Solutions of sodium thiosulphate, cyanide, sodium sulphide,

\* Abstract of a Paper read before the Royal Society, March 17, 1904.

Dr. STEINHART did not agree with the "strain" theory put forward by the author. All materials contain some trace of saline matter, and one is almost sure to get chlorine or some such substance given off on electrolysis.

Mr. DARLING, in reply, thanked the speakers for their useful suggestions, many of which he had not had time to follow up, but hoped to in the future. Referring to Mr. Bluman's remarks, organic substances eventually discoloured the paper. He did not think hydrogen the cause of the latent images; certainly, hydrogen peroxide does not destroy the image.

## NOTICES OF BOOKS

*The Principles of Leather Manufacture.* By H. R. PROCTER, F.I.C., F.C.S. London: E. and F. N. Spon, Limited. New York: Spon and Chamberlain. 1903. Pp. 512.

THE present volume is intended not only for the chemist, but also for the practical tanner, though we might well ask why these two capacities should not be combined in one man. Surely, in these days of technical education, there cannot be many "practical tanners" who are not also chemists, while the proper control of the putrefactive and fermentation processes must also involve more than a superficial acquaintance with bacteriology. But whether the reader be one or the other he will doubtless benefit by a study of these pages.

In the twenty-eight chapters into which this book is divided, the author takes us through the history and practice of tanning and leather manufacture from the early primitive methods used by the ancients to the latest processes in vogue, and finally gives us a chapter on waste products and their disposal, including the bacterial purification of sewage. The book is illustrated with over 100 cuts of machinery and apparatus, including a number of microphotographs, these being very interesting. There is also a good index of fourteen pages.

*Metallurgical Analysis and Assaying. A Three Years' Course for Students of Schools of Mines.* By W. A. MACLEOD, B.A., B.Sc., A.O.S.M. (N. Z.), and CHARLES WALKER, F.C.S. With 109 Figures in the Text. London: Charles Griffin and Company, Limited. 1903. Pp. 318.

ACCORDING to the opinions expressed by the authors, the time necessary to learn metallurgical analysis and assaying is 231 hours in the first year, 495 hours in the second year, and 792 hours in the third. This progressive increase in time seems considerable, but when looked at closely does not amount to many hours per week. For instance, in the third year, which is devoted to assaying and technical analysis, only 24 hours per week for thirty-three weeks is allotted, or four hours a day; this is surely very little. In our student days the metallurgical laboratory was open for eight hours daily, less three lectures per week (of one hour each) for metallurgical students, and we generally had lunch in the laboratory, or, at most, took half-an-hour off for the purpose. However, we will let that pass and come to the methods of instruction proposed.

Naturally, the student commences with chemistry pure and simple, and in Part I. will find instruction in simple qualitative analysis, the preparation and properties of gases, and the group reactions, &c.

Part II. completes the scheme of qualitative analysis, and deals with quantitative analysis by the gravimetric, volumetric, colorimetric, and electrolytic methods.

In Part III., divided into two sections, we come to assaying and technical analysis. Assaying, which may be defined as "the estimation of the commercially important elements in the ores, alloys, or products," is classified into the dry or fire-assay method and the wet way, the latter

including those methods which have been learnt in Part II. The first six chapters in the section on fire-assay methods deal with descriptions of the reagents used, the fitting-up of the assay laboratory, manipulation, the preliminary examination of ores, and introductory experimental work. Then we come to the specific assay of the different metals—tin, lead, gold, silver, copper, sulphur, and mercury; in most cases a separate chapter being devoted to each metal.

The section on Technical Analysis deals with those substances which are connected with metallurgy and metallurgical methods, such as water analysis, the analysis of furnace gases, coal, coke, fire-clays, cements, and slags, and the analysis of iron and steel.

The whole volume is well arranged and written, and although it cannot be claimed that there is anything especially new in the methods described, there is ample evidence of the care with which the compilation has been made, combined with a thorough knowledge of the technical details involved in successfully carrying them out.

We can confidently recommend this book to students, and also to those in practice, who will find it useful for reference. The index is rather scanty, but this may be improved in a future edition.

*Elements of Inorganic Chemistry.* By HARRY C. JONES. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1903. Pp. 343.

THE author is of the opinion that some of the generalisations of the new physical chemistry can—and ought to be—introduced very early in the study of the science. Chemistry has changed very much in the last twenty years, and we quite agree that some change should be made in the manner of teaching it. He refers in the preface to a typical case, viz., law of electrolytic dissociation, which has been firmly established, by which it is shown that it is the ions and not the atoms that are the active agents chemically. If the student has been taught the contrary in the early stages of his work, he will have to unlearn it later on.

In this volume the physical properties of substances are treated rather more fully than is usual in books for beginners, since the tendency is to bring chemistry and physics closer together.

The book is divided into twenty-seven chapters, the first twelve being of a general character. Chapter XIII. is on the Periodic Law; and, beginning with Chapter XIV., the elements are taken for consideration one by one, and their reactions fully described.

The proofs might have been corrected with more care, as there are not a few misprints. For instance, in the list of elements praseodymium is spelt with an *o* for the *a*, and thallium with one *l*. But this does not detract seriously from the merits of the book, which is well written and arranged. There is a good index covering seventeen pages.

*Karl Heumann's Anleitung zum Experimentieren bei Vorlesungen über Anorganische Chemie.* ("Karl Heumann's Directions for Lecture Experiments in Inorganic Chemistry"). By Prof. Dr. O. KÜHLING. Braunschweig: Friedrich Vieweg und Sohn. 1904.

THE author of this work died some ten years ago, soon after the appearance of the second edition, and Professor Kühling, recognising the great value of the work for technical schools and universities, has brought out this third edition. The progress of many branches of inorganic chemistry has necessitated a certain amount of alteration in the text; for instance, space has had to be made for the addition of many important experiments in electrochemistry, and others performed with the assistance of great extremes of temperature, with liquid air, &c., and this has been done by omitting repetitions of various methods of the preparation of the same compound, or of the preparation of compounds which can be bought very cheaply; these omissions have not at all impaired the usefulness of

the book. The introduction will be found specially valuable in the hints and suggestions it gives to demonstrators and assistants. Much of the information concerning such matters as the handling and use of electrical and lantern apparatus is not to be found in ordinary textbooks of practical chemistry, and will be very acceptable. The descriptions of the experiments, though covering only very familiar ground, are conveniently arranged, and perfectly simple and practical. A very short account of the radio-activity of certain substances is interesting and fairly complete; this has been mainly supplied by Prof. Marckwald, and bears the impress of his views; as, for example, in speaking of radio-tellurium as a distinct new substance.

*Zeitschrift für Physikalische Chemie.* Sechshundvierzigster Band. ("Journal of Physical Chemistry." Vol. XLVI.). Edited by WILH. OSTWALD and J. H. VAN'T HOFF. Leipzig: Wilhelm Engelmann. 1903.

THIS volume of the *Zeitschrift für Physikalische Chemie* is a "Jubiläum" for Prof. Ostwald in celebration of his jubilee. It contains as frontispiece an excellent portrait of the great chemist, and an introduction by Prof. J. H. van't Hoff, in which a short biographical sketch is given with an appreciation of Ostwald's work as an investigator, thinker, teacher, organiser, and reformer, which is very interesting reading. In so short a space it is not possible to do more than indicate the lines of his activity in these respects, or to make more than passing allusion to the monumental work, the "Lehrbuch der Allgemeinen Chemie," but the author of the sketch certainly does not give the impression of under-estimating the great importance of Ostwald's work in any region. A list of his writings is sufficient to show his extraordinarily great literary activity. The papers contained in the volume come from the pens of Ostwald's pupils from many parts of the world, and relate to different branches of physical chemistry. German, English, and French workers have united to honour the great master, of whom it is not too much to say that mankind owes him an ever-increasing debt.

## CORRESPONDENCE.

### CATALOGUES GRATIS.

*To the Editor of the Chemical News.*

SIR,—Taking it for granted that an invitation—prominent in the *CHEMICAL NEWS*, and editorially recommended—was extended to all subscribers, whatever air we might breathe, I wrote for a copy of the book in question, and in due time received the enclosed response, which one might feel disposed to deem amusingly discourteous.

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That Becker would have received no order from me is evident. Home markets supply all our needs. For such is the effect of the high tariff to which he takes exception: it teaches a people to produce whatever they cannot advantageously import. And Americans are not unapt: balances rivaling Becker's are now manufactured in Denver, Colorado, a city which, I doubt not, most people within sound of Bow bells, if they know at all, consider a cowboy's metropolis.

Nevertheless, having publicly offered his Catalogue gratis to the world at large, he should honour all demands. He should murmur with Martial, *ad librum suum*, "I, fuge, sed poteras tutor esse domi," and send his little book freely forth into the unappreciative world.—I am, &c.,

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## AUSTRALIAN PATENT LAW.

*To the Editor of the Chemical News.*

SIR,—Your readers will probably be interested in hearing that the Governor-General in Council of Australia has decreed, in accordance with the new Federal law, that patent applications for Australia can now be formally made at the Custom House of the capital city of each state. Applications so filed will be marked with the date, hour, and minute of receipt, and the applications will be subsequently recorded at the Patent Office as having been filed at that date. Applications thus filed will not be merely taken in order of their dates, but they will be held to be dated as far as regards novelty from the date of actually filing at the Custom House, unless dated under the convention. Under the convention nearly all the principal countries and colonies have agreed to grant the right to a patentee who has filed an application for a patent in one of the countries or colonies to date any or all the other applications he may make for the same invention in other realms during the year, as of the date of his original first application. The application in Australia can either be a provisional one or a complete. The fees and stamp duties are just double those of an application for Great Britain and Ireland.—We are, &c.,

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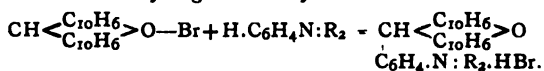
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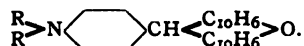
*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 9, February 29, 1904.

**Cadmium Arsenide.**—Albert Granger.—When cadmium is heated in arsenic vapour in presence of hydrogen or another inert gas, the two elements easily combine. The cadmium arsenide is in the form of reddish crystals. Its density at 15° is 6.211, and its composition is  $Cd_3As_2$ . It corresponds to cadmium phosphide,  $Cd_3P_2$ . This arsenide is a much more stable substance than copper arsenide. Its action on acids and the halogens is that of arsenides in general. It is soluble in nitric acid even when diluted with water. It is also attacked by chlorine, bromine, and all chloro-mixtures, as aqua-regia besides, also, oxidising mixtures.

**Union of Dinaphthopyryl Salts with Di-alcoholic Aromatic Amines.**—R. Fosse.—Salts of dinaphthopyryl react on the dialcoholic aromatic amines to give new bases, resulting from the substitution of the dinaphthopyryl radical for an atom of hydrogen of the cyclic amine :—



The union of the dinaphthopyryl radical takes place with the benzenic carbon of the amine in the para-position, according to the formula—



The author establishes, by synthesis, the constitution of one of these products of union. The body resulting from the action of dinaphthopyryl bromide on dimethylaniline is identical with the product of condensation of dimethylpara-aminobenzaldehyde and  $\beta$ -naphthol.

**Ethylidene Camphor.** Ethylhomocamphoric Acid.—J. Minguin.—The *al*-camphors of the aromatic series, as M. Haller has shown, have a rotatory power considerably higher than the corresponding alcoyl camphors. The author examines ethylidene camphor—the superior homologue of the methylenic derivative—with regard to its rotatory power, and finds that this has also a much greater value than the corresponding *al*-camphor.

**Synthesis of  $\alpha\alpha$ -Dimethylglutaric and  $\alpha\alpha$ -Dimethyladipic Acids.**—G. Blanc.—The author easily synthesises  $\alpha\alpha$ -dimethylglutaric and  $\alpha\alpha$ -dimethyladipic acids by applying the general method of synthesis of the alcohols described by the author and M. Bouveault. The formation of these two acids, starting from the lactones (themselves obtained by the reduction of  $\alpha\alpha$ -dimethylsuccinic and  $\alpha\alpha$ -dimethylglutaric ethers), prove that the reduction takes place on the carboxyl united to the primary carbon. The author succeeds in generalising this law.

## MISCELLANEOUS.

**The Paper Trade Directory for 1904.**—This edition, edited as usual by "The Doctor," who is the editor of *Paper Making*, includes the names and address of all our Colonial and Indian paper and pulp mills, besides those of Great Britain. Every care has been taken to correct, revise, and bring up to date the information herein given. Three mills have been closed and dismantled during the past year, five have changed hands, and several new paper machines have been put to work in the place of older and smaller machines. We are glad to see that the paper trade is in a fairly prosperous condition.

**Ventilation of the House of Commons.**—The report of the Select Committee on Ventilation appointed by the House of Commons has been recently issued, and is now reprinted in the form of a pamphlet by Messrs. Hickson, Ward, and Co. It appears from a perusal of this report that the general consensus of opinion condemns mechanical ventilation by means of fans. No matter what precautions are taken, it is impossible to avoid draughts, and even then members of the House complain of tiredness and lassitude in spite of the apparent purity of the air as shown by analysis. It is generally agreed that ventilation can only be achieved successfully by natural means as opposed to mechanical methods.

**Percarbonate of Sodium.**—S. Tanatar.—The author has already described percarbonate of sodium,  $\text{Na}_2\text{CO}_4 + \frac{1}{2}\text{H}_2\text{O}$ , obtained by the action of  $\text{H}_2\text{O}_2$  on a solution of  $\text{Na}_2\text{CO}_3$ . In aqueous solution this salt gradually decomposes into  $\text{Na}_2\text{CO}_3$  and  $\text{H}_2\text{O}_2$ ; thermochemical determinations have shown that the solution of this salt in water is different to a mixture of solutions of  $\text{Na}_2\text{CO}_3$  and  $\text{H}_2\text{O}_2$ ; only, as we have not obtained any percarbonates of the heavy metals by the reciprocal action

of  $\text{Na}_2\text{CO}_3$  and the salts of those metals, we may ask whether the percarbonates are not carbonates in which the  $\text{H}_2\text{O}_2$  has replaced the water of crystallisation. The discovery of certain compounds ( $\text{KF} + \text{H}_2\text{O}_2$ ;  $\text{NaSO}_4 + \text{H}_2\text{O}_2 + 9\text{H}_2\text{O}$ ), seems to confirm this idea. In an attempt to solve this question, the author has endeavoured to find the coefficient for the division of  $\text{H}_2\text{O}_2$  between water and ether, then between a solution of  $\text{Na}_2\text{CO}_3$  and ether; he found that by the addition of  $\text{Na}_2\text{CO}_3$ , about 30 per cent of the possible quantity of  $\text{H}_2\text{O}_2$  unites with the salt. Thus percarbonate of sodium in solution is only partially decomposed into  $\text{Na}_2\text{CO}_3$  and  $\text{H}_2\text{O}_2$ . Further, many of the salts of the hyperacids behave in this manner. The rise of temperature and the diminution of the concentration increases the hydrolysis. Though these facts may not be sufficient to draw accurate conclusions, the author thinks that percarbonic acid, and the hyperacids in general, contain true higher oxides of the elements.—*Journ. Soc. Phys. Chim. R.*, vol. xxxiv., p. 952.

**A New Method for the Quantitative Separation of Tellurium and Antimony.**—A. Gutbier.—I. *Separation by means of Hydrate of Hydrazine.*—The solution should be a hydrochloric one, and free from sulphuric and nitric acids. To the slightly acid solution we add a large excess of tartaric acid and an excess of a dilute solution of hydrate of hydrazine. The tellurium is precipitated; the antimony remains in solution. The latter is boiled with concentrated hydrochloric acid, diluted with warm water, and precipitated with sulphuretted hydrogen. Instead of the hydrate of hydrazine, we might use the hydrochlorate, but not the sulphate. The separation is quite general. The antimony and the tellurium might be precipitated first of all as sulphides, and these subsequently oxidised with nitric acid or aqua regia. II. *Separation by means of Hydrochlorate of Hydroxylamine* (in collaboration with F. Resenscheck).—This separation, already suggested by Jannasch and Müller (*Berichte*, vol. xxxi., p. 2338), is effected in a strongly ammoniacal solution. The solution containing the chlorides is treated with a large excess of tartaric acid, and then with 1 or 2 grms. of solid hydrochlorate of hydroxylamine. When the precipitate has settled, it is filtered, and the antimony is estimated in the solution as above. It must first be ascertained that the whole of the tellurium has been precipitated. The two methods give good results, but we do not strongly recommend the precipitation by hydrochlorate of hydroxylamine, on account of the loss of time it occasions.—*Zeit. Anorg. Chem.*, vol. xxxii., p. 260.

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*Manuals:* E. Chr. Hansen, "Practical Studies in Fermentation," London (Spon), 1896. Alfred Jörgensen, "Micro-Organisms and Fermentation," London and New York (Macmillan and Co.), 1900.

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### CONTENTS.

| ARTICLES:—                                                                                                                                                                                                                                                        | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Liquid State, by P. J. Beveridge, M.A., B.Sc.                                                                                                                                                                                                                 | 169  |
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S.                                                                                                                                                                                       | 170  |
| Contributions to the Chemistry of the Rare Earths, by Chas Baskerville and Eugene G. Moss                                                                                                                                                                         | 172  |
| Eleventh Annual Report of the Committee on Atomic Weights—Determinations Published during 1903, by F. W. Clarke                                                                                                                                                   | 173  |
| PROCEEDINGS OF SOCIETIES—                                                                                                                                                                                                                                         |      |
| CHEMICAL SOCIETY.—Mercuric Nitrite and its Decomposition by Heat—Note on the Higher Glycerides—Isomeric Change of Diacylanilides into Acyl-amino-ketones, Transformation of the [1-Dibenzoyl] Toluidines into the Isomeric Methyl-benzoyl-amino-benzophenones—&c. | 175  |
| PHYSICAL SOCIETY                                                                                                                                                                                                                                                  | 177  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                                                                                                                                             | 179  |
| MISCELLANEOUS                                                                                                                                                                                                                                                     | 179  |
| MEETINGS FOR THE WEEK                                                                                                                                                                                                                                             | 180  |

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Fellow and Prælector in Chemistry of Gonville and Caius College,  
Cambridge.

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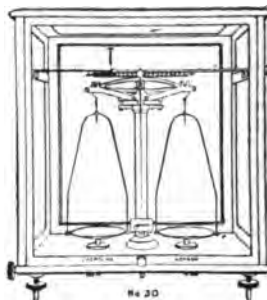
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2315.

## THE LIQUID STATE.

By P. J. BEVERIDGE, M.A., B.Sc.

WHEN we consider the astonishing resemblance that exists between the nature of gases, as explained by the kinetic theory and the condition of dissolved molecules, the quantitative identity between the gas equation and the equation for osmotic pressure, is it not justifiable to make a tentative application of the kinetic properties of the molecule beyond the case of a mere dilute solute to attempt a general application to the liquid state? The remark is as old as Graham that a solute takes upon itself the liquid character. Is it, then, philosophical to shrink from applying the mechanical properties of the dissolved molecule to the whole of the liquid molecules present, to draw a hard and fast line at dissolved substances, to protest with Pattison Muir that the term molecule can be applied to liquids only in a vague manner and without strict quantitative significance (Watts' Dictionary, Article "Atomic and Molecular Weights"), and not at least to inquire whether the principles which van't Hoff has deduced from osmosis are not applicable to all molecules that are in the liquid condition; in short, to attempt a far reaching generalisation of liquid and gas? It is here intended to inquire whether such an extension of the kinetic theory is possible and congruous with known facts.

The kinetic theory gives us the gas equation,  $pv = Rt$ , expressing *in parvo* the physical properties of a perfect gas. If we are dealing with molecular quantities (1 grm.-mol.)  $pv = 84700t$  in mechanical measure, or  $pv = 2t$  (more accurately 1.99t) in thermal measure. From this equation is deduced Avogadro's law, and from it also may be shown that the translatory energy of a grm.-mol. of any gas is 3t (or 2.99t).

Let us then inquire whether provisionally we may consistently assume, first, that the equation  $pv = 2t$  can be applied to liquid molecules; secondly, that Avogadro's law holds good in the liquid state; thirdly, that the translatory kinetic energy of the liquid molecule is the same as of the gas molecule at the same temperature; lastly, that the gas molecules in passing into the liquid condition are not greatly, if at all, polymerised. All these assumptions are largely interdependent, and a proof of any one of them goes towards the establishment of the whole.

To consider first of all the question of polymerisation; for unless we have a measure of the molecular weight all further discussion is futile. It is commonly assumed that liquid molecules are very largely polymerised, that in fact they are many times greater than the gas molecules; how many times greater we have no means to judge. On what grounds can we be justified in asserting, on the contrary, that the liquid molecule is practically the same as the molecule of the gas?

In the first place, there is Trouton's law, a numerical statement regarding the heat of vaporisation, which holds good with wonderfully little variation for all liquids. It may be stated thus:—The molecular heat of vaporisation of a liquid boiling at atmospheric pressure, that is the heat required to produce 1 grm.-molecular weight of gas from a liquid at its boiling-point ( $t^\circ$  abs.), is equal to about 19t calories (+2t for external work on atmospheric pressure). This law has an accuracy beyond most empirical laws of physics; it is quite comparable in this respect with Dulong and Petit's law, which is so fundamental to the atomic theory. Now, either the liquid molecules are invariable multiples of the gas molecule or they are not. On the one hand, the supposition that the liquid molecule is always just  $n$  times the gas molecule ( $n$  greater than unity) is hardly conceivable in view of the exceedingly irregular character of molecular polymerisation when it does occur.

On the other hand, suppose we have various liquids whose molecules are respectively made up from, say, two, five, fifty gas molecules, it is simply impossible to suppose that the same amount of heat is required not only to effect the change of physical state but also to dissociate these unequal polymerisations. The only admissible explanation therefore is that liquid molecules are as nearly as possible identical with their gas molecules.

And take the case of acetic acid. Its vapour density indicates a partial polymerisation at boiling-point. But the heat of vaporisation corresponds to the experimental, not to the theoretical molecular weight. Could there be clearer proof that the liquid molecule is polymerised in an equal degree to the gas molecule?

Again, another argument may be deduced from the reduction of the vapour tension of a solvent. The law may be stated thus:—

Vapour pressure of pure solvent

Decrease of pressure in solution

$$= \frac{\text{Total number of molecules, solvent and solute}}{\text{Number molecules of solute}}$$

Or if  $p, p'$  be the pressures,  $N$  the number of molecules of solvent present in the solution,  $n$  the number of molecules of solute—

$$\frac{p}{p - p'} = \frac{N + n}{n}$$

In other words, the decrease of the pressure is proportional to the number of molecules of the volatile solvent which may be supposed exchanged for molecules of non-volatile solute. Given  $p, p'$ , and  $N$  it is admitted on all hands that we may calculate  $n$  the number of molecules of solute, and hence also the molecular weight of the solute. But, conversely, when we are dealing with a known number of molecules of solute and propose to calculate thence  $N$  and the molecular weight of the solvent, then at once we receive elaborate cautions on all sides, we are told that the result will not give the molecular weight of the liquid solvent, but the molecular weight of the corresponding gas (Arrhenius, "Text-book of Electro-chemistry," English translation, p. 42). I for my part fail to see how any experiment on a change of condition in the liquid could work out the molecular weight of its gas if the liquid and gas molecules were not identical. And when Arrhenius (*loc. cit.*) triumphantly points out that such an experiment with acetic acid as a solvent will work out to give the mean molecular weight of the partially polymerised acetic acid gas at the temperature of working, one can only reply that there is hardly need of any stronger proof that the gas and liquid molecules are identical.

Polymerisation may indeed be to some extent a characteristic of the change from gas to liquid, but the extent is generally exaggerated. There is strong evidence of polymerisation in the molecules of a solute with increasing concentration, but it will be found that this takes place not so much when the increasing concentration means an approach to the separation or gradual purification of a dissolved liquid, but when approach is being made to the crystallising out of a solid. Thus sulphur in carbon disulphide shows molecular weight as high as  $S_8$  (indeed, I have even reached  $S_{12}$ ), but if it is a liquid that is in solution, for example, turpentine dissolved in ether, the signs of polymerisation are by no means so conclusive. And even in the case of the solution of solids, the proper way to regard these vapour tension experiments is not to declare that the formula fails for high concentrations, but to admit that even at high concentrations it gives with practical accuracy the average molecular weight of the highly polymerised molecules.

Indeed, if it were not that there seems to be some hazy idea that we are fishing in liquid solutions for the gas-molecular weights of the solutes, the coincidence between the gas-molecular weights of vaporisable substances and their molecular weights in solution would have settled the question long ago.

(To be continued.)

## THE ELEMENTS: VERIFIED AND UNVERIFIED.

By CHARLES BASKERVILLE, Ph.D., F.C.S., Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 163).

## LIST OF PROPOSED CHEMICAL ELEMENTS (continued).

| Date.                                            | Name.                                                                              | Source.                                                  | Authority.                                                     | Reference.                                                                                                                                 | Remarks.                                                                                                                                                                                                                                                                                                                                                                                                     |
|--------------------------------------------------|------------------------------------------------------------------------------------|----------------------------------------------------------|----------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1877<br>1878<br>1878                             | Lavoesium.<br>"New earths."<br>"X."                                                | Pyrite.<br>Unnamed mineral.<br>Gadolinite.               | Prat.<br>Gerland.<br>Soret.                                    | Le Monde Pharmaceutique.<br>Chemical News, 38, 136.<br>Comptes Rendus, 88, 106a.                                                           | Identical with holmium subsequently discovered by Cleve, giving bands $\lambda$ 640 and 536.3 with $\lambda$ 451.5 and 753. More soluble formate. Characterised by absorption band $\lambda$ 450 (dysprosium in Soret's "X"). Roscoe (Ber., 1882, 75, 1274), showed it in a mixture of terbia and yttria. Also denied by Crookes (Phil. Trans., 174, 910) and Urbain (Ann. Chim. Phys., 1900, 171, 19, 192). |
| 1878                                             | Philippium.                                                                        | Samarските.                                              | Delafontaine.                                                  | Comptes Rendus, 87, 559;<br>Chem. News, 36, 202.                                                                                           | Absorption bands $\lambda$ 416 and $\lambda$ 478—one now belonging to samarium of de Boisbaudran—would be decipium now, but Delafontaine said his had no absorption bands. While trying to isolate Delafontaine's philippium.                                                                                                                                                                                |
| 1878                                             | Decipium.                                                                          | Samarските.                                              | Delafontaine.                                                  | Comptes Rendus, 87, 632;<br>Chem. News, 38, 223.                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                              |
| 1878                                             | Ytterbium.                                                                         | Gadolinite.                                              | Maignac.                                                       | Comptes Rendus, 87, 578;<br>Chem. News, 38, 213.                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                              |
| 1879                                             | Scandium.                                                                          | Gadolinite.                                              | Nilson.                                                        | Comptes Rendus, 88, 645;<br>Chem. News, 40, 76; Ber., 12, 554; 13, 1439.                                                                   | Definite spark spectrum (Thalén, Comptes Rendus, 81, 451; 88, 646; Chemical News, 47, 217).                                                                                                                                                                                                                                                                                                                  |
| 1879                                             | Norwegium.                                                                         | Gersdorffite.                                            | Dahl.                                                          | Zeit. d. Geol. Gesell., 31, 480; Comptes Rendus, 89, 47; Chem. News, 40, 25.                                                               | Atomic weight ascribed 145.9. Not enough had for work. Mendeleeff hoped that it would prove to be eka-cadmium. Brauner suggested Ng = 219; not proven. Delafontaine's original decipium. Demarcay separated europium from it.                                                                                                                                                                                |
| 1879                                             | Samarium.                                                                          | Samarските.                                              | De Boisbaudran.                                                | Comptes Rendus, 88, 322;<br>Chem. News, 40, 99.                                                                                            | Misapprehension.                                                                                                                                                                                                                                                                                                                                                                                             |
| 1879                                             | Barcenium.                                                                         |                                                          |                                                                | Wagner's Jahresbericht, 189, 8.                                                                                                            | In examining Mosander's erbia.                                                                                                                                                                                                                                                                                                                                                                               |
| 1879<br>1879<br>1879                             | Thulium.<br>Holmium.                                                               | Gadolinite.<br>Gadolinite.                               | Cleve.<br>Cleve.                                               | Comptes Rendus, 89, 478.<br>Comptes Rendus, 89, 478.                                                                                       | Admitted by Cleve to be Soret's "X." Subsequently found by de Boisbaudran to be a mixture of true holmium and dysprosium (Comptes Rendus, 1886, 102, 1003). Crookes says only one of the several bands, 451.5, belongs to dysprosia.                                                                                                                                                                         |
| 1879                                             | Ouralium.                                                                          | Russian platinum.                                        | Guyard.                                                        | Monit. Scient., [3], 9, 795;<br>Chem. News, 40, 57.                                                                                        | Resembles platinum, but softer; atomic weight 187.5. Gives orange melt with potassium cyanide. Unverified. Not to be mistaken for columbium (niobium).                                                                                                                                                                                                                                                       |
| 1879                                             | Columbium.<br>Rogerium.<br>Vesbium.                                                | Samarските.<br>Samarските.<br>Lava (Vesuvius).           | J. L. Smith.<br>J. L. Smith.<br>Scacchi.                       | Chem. News, 41, 116.                                                                                                                       | Proved to be vanadium oxide. <i>Vide</i> Rammelsberg (Ber., 1880, 13, 250).                                                                                                                                                                                                                                                                                                                                  |
| 1880<br>1880<br>(1886)<br>1880<br>(1885)<br>1881 | Comesium.<br>Y <sub>a</sub> —GADOLINIUM.<br>Y <sub>2</sub> —SAMARIUM.<br>Actinium. | Samarските.<br>Samarските.<br>Samarските.<br>Zinc white. | Kammerer.<br>Maignac.<br>Maignac.<br>Delafontaine.<br>Phipson. | Chem. Zeit., 1880, 273.<br>Arch. des Sci. Phys. et Nat., 131, 3, 413; Comptes Rendus, April, 104, No. 16.<br>Chem. News, 44, 73, 138, 191. | These later proved to be complex, but definite elements by the second names had from them. Delafontaine decomposed didymium, which proved to be identical with Y <sub>c</sub> , Delafontaine's original decipium.                                                                                                                                                                                            |

|      |                                            |                                                                         |                 |                                                        |                                                                                                                                                                                                                                                                                                                                                                        |
|------|--------------------------------------------|-------------------------------------------------------------------------|-----------------|--------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1882 | Di?                                        | Gadolinite.                                                             | Cleve.          | Chem. News, 45, 273.                                   | Element with ignition spectrum ray $\lambda$ 4333 $\mu$ . Found in number of minerals.                                                                                                                                                                                                                                                                                 |
| 1883 | Nameless.                                  | Platinum ore.                                                           | Wilm.           | Berichte, 16, 1298.                                    |                                                                                                                                                                                                                                                                                                                                                                        |
| 1884 | Idunium.                                   | Vanadium ore.                                                           | Websky.         | Sitzungsab. Ak. Wiss., Berlin, 30, 661.                |                                                                                                                                                                                                                                                                                                                                                                        |
| 1885 | NEODIDYMIUM.                               | Didymium.                                                               | Auer.           | Monats., 6; Chem. News, 52, 49.                        | "Meta-elements," no doubt of complexity. See work of Krüss, Nilson, Kieselwetter, Brauner, Crookes, Bettendorff, Forthing, Thompson, Dennis, Boudouard, and Baskerville.                                                                                                                                                                                               |
| 1885 | PRASEODIDYMIUM.                            | Didymium.                                                               | Auer.           | Monats., 6; Chem. News, 52, 49.                        | Identical with Crookes's Gd. Probably terbium, apparently identical with Crookes's Gf. See Crookes below.                                                                                                                                                                                                                                                              |
| 1885 | Za.<br>Zd.<br>Zy.                          | Didymium.                                                               | De Boisbaudran. | Comptes Rendus, 102, 153 and 1887; Chem. News, 53, 69. |                                                                                                                                                                                                                                                                                                                                                                        |
| 1885 | Unnamed.                                   | Cerite.                                                                 | Brauner.        | Journ. Chem. Soc., 47, 879.                            | Brauner (Monatshefte, 3, 487) didymium $\beta$ and perhaps didymium $\gamma$ .                                                                                                                                                                                                                                                                                         |
| 1886 | Zy.                                        | Terbia.                                                                 | De Boisbaudran. | Comptes Rendus, 152, 103; Chem. News, 53, 63.          | By electric spectrum lines $\lambda$ 583.5, 575, 570, 526.9.                                                                                                                                                                                                                                                                                                           |
| 1886 | Dysprosium.                                | Soret's "X" (holmium).                                                  | De Boisbaudran. | Comptes Rendus, 102, 1003.                             | Band $\lambda$ 451.5 and $\lambda$ 752.                                                                                                                                                                                                                                                                                                                                |
| 1886 | Dysprosium X.                              | Dysprosium.                                                             | Crookes.        | Proc. Roy. Soc., 40, 502.                              | Having absorption $\lambda$ 451.5 only. Agreed to by Boisbaudran. (See Holmium).                                                                                                                                                                                                                                                                                       |
| 1886 | Unknown.                                   |                                                                         | Crookes.        | Proc. Roy. Soc., 40, 502.                              | Absorption band 550 and 493 no: in thulium, erbium, holmium, and dysprosium.                                                                                                                                                                                                                                                                                           |
| 1886 | Austrum.                                   | Orthite.                                                                | Linnemann.      | Nature, 34, 59; Monatshefte, 1886, 773.                | Orthite re-examined by Pribram (Monats., 1900, 21, 148), who confirms de Boisbaudran's assertion (Comptes Rendus, 1886, 102, 1436) that this "austrum" is identical with gallium.                                                                                                                                                                                      |
| 1886 | GERMANIUM.                                 | Argyrodite.                                                             | Winkler.        | Berichte, 19, 210; Chemical News, 54, 136.             | As predicted by Mendeleeff.                                                                                                                                                                                                                                                                                                                                            |
| 1886 | Da.<br>Sb.<br>Sy.<br>Ga.<br>Gb.            | Didymium.<br>Samarskite.<br>Samarskite.<br>Gadolinite.<br>Gadolinite.   | Crookes.        | Chemical News, 54, 13; Nature, 34, 166.                | New.<br>New.<br>Ytterbium.<br>New.<br>Based upon radiant matter spectroscopy. Strongly opposed by de Boisbaudran (Chemical News, 54, 16). Truly "a nebula of elementary matter," as Petterson put it. See original for vacuum band belonging to each.                                                                                                                  |
|      | Gy.<br>Gb.<br>Gc.<br>Gd.<br>Ge.            | Gadolinite.<br>Gadolinite.<br>Gadolinite.<br>Gadolinite.<br>Gadolinite. |                 |                                                        | Gadolinium or Zb. See de Boisbaudran (Comptes Rendus, 103, 114).<br>New.<br>New, or Za (see de Boisbaudran).<br>New.<br>New.<br>New.                                                                                                                                                                                                                                   |
| 1886 | Polymestrum.<br>Nameless (1).<br>Erebodum. | Glacial debris in Scotland.                                             | Pringle.        | Chemical News, 54, 167.                                | One must read the original to appreciate the properties of these elements, of which we have heard nothing since their announcement. Considering these facts, the country of the discoverer, and the time of the presentation of the paper (during the heat of the Crookes-de Boisbaudran controversy) without inside information, an American might appreciate a joke. |
|      | Nameless (2).<br>Gadenium.<br>Hesperisium. |                                                                         |                 |                                                        |                                                                                                                                                                                                                                                                                                                                                                        |

(To be continued).

CONTRIBUTIONS TO THE CHEMISTRY OF THE  
RARE EARTHS.\*

## ON LANTHAN-ALUMS—SOME NEW DOUBLE SULPHATES.

By CHAS. BASKERVILLE and EUGENE G. MOSS.

**Synopsis.**—This paper gives an account of some attempts, not altogether successful, to prepare lanthan-alums. The characteristic properties of lanthanum sulphate, namely, ready formation of a comparatively insoluble hydrate and double salts, appear to interfere. The following new salts, belonging to known types, were prepared:— $\text{La}_2(\text{SO}_4)_3 \cdot \text{Rb}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$  and  $\text{La}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ , and  $\text{La}_2(\text{SO}_4)_3 \cdot \text{Rb}_2\text{SO}_4$ , as well as another model, viz.,  $3\text{La}_2(\text{SO}_4)_3 \cdot 2\text{Rb}_2\text{SO}_4$  and  $3\text{La}_2(\text{SO}_4)_3 \cdot 2\text{Cs}_2\text{SO}_4$ .

It may be recalled that lanthanum was one of the elements whose atomic weight was corrected by Mendeleëff in the enunciation of the periodic law ("Principles of Chemistry" [English translation], vol. ii., p. 88). Lanthanum is now placed in the aluminum group. Many of the sulphates of elements which form sesquioxides, form the basis of the large class of bodies known as the alums. For these two reasons it is natural to expect lanthanum to form an alum.

Locke (*Am. Chem. Journ.*, xxvii., 281) has called attention to the fact that if an element will form an alum, it does so more readily with caesium sulphate than with any of the other alkaline sulphates. Experiments were therefore begun with lanthanum sulphate and caesium sulphate in an equimolecular mixture. In endeavouring to concentrate the solution to the crystallisation point by heating, either directly or by evaporation upon the water-bath, there resulted almost immediately a precipitation of the nonhydrated lanthanum sulphate,  $\text{La}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$ . It is well known that lanthanum sulphate dehydrates in this manner. We learn also that this body fails to go back into the solution in the same amount of water on cooling. Muthmann and Rügig, in their investigation on the separation of the cerite metals through the solubility of their sulphates, state that the solubility of this nonhydrate varies but slightly with the temperature (*Ber.*, 1898, xxxi., 1723).

As it was impossible to bring about the proper concentration of the mixture by heating, it was placed in a vacuum desiccator and allowed to evaporate under diminished pressure. Beautiful large crystals, symmetrical in form, separated out. The analysis of these crystals, from which the mother-liquor was drained, gave the following percentages:—

|                                                                                         | Lanthanum oxide. | Cæsium oxide. |
|-----------------------------------------------------------------------------------------|------------------|---------------|
| Calculated for—                                                                         |                  |               |
| $\text{La}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ .. | 23.85            | 20.76         |
| $\text{La}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4$ .. ..                         | 35.13            | 30.39         |
| $3\text{La}_2(\text{SO}_4)_3 \cdot 2\text{Cs}_2\text{SO}_4$ .. ..                       | 37.57            | 21.60         |
| Found .. .. .                                                                           | 33.75            | 21.72         |

Another experiment was carried out by placing a similar solution in a freezing-mixture of ice and salt, but no crystals were formed.

During the progress of these experiments, efforts were made to prepare lanthan-potash alum by electrolytic oxidation. Howe and O'Neal prepared iron, cobalt, and chromium alums and attempted to make manganese alums by electrolysis, but without success (*Journ. Am. Chem. Soc.*, 1898, xx., 764). Later Piccini prepared the caesium manganese alum by that method (*Zeit. Anorg. Chem.*, 1899, xx., 12). This method, varied in some respects, was used with lanthanum.

A saturated solution of lanthanum sulphate was prepared by saturating sulphuric acid with lanthanum hydroxide, which had been obtained by precipitation from lanthanum ammonium nitrate. The solution of lanthanum

sulphate was placed within an unglazed porcelain annealing dish. This rested in a large beaker containing more than enough double sulphate of potassium and lanthanum to make a saturated solution. It is difficultly soluble in water, hence the excess of solid. A spiral platinum cathode was placed in the solution in the annealing dish, while a sheet platinum anode rested in the outer liquid. Four Lalande-Edison cells were used, giving a current of about 0.16 ampère, and the process was allowed to continue sixteen hours. No perceptible change in either the outer or inner solution was noted. A few white, opaque crystals separated about the top of the annealing cup. The solution within the annealing cup was taken out and allowed to evaporate spontaneously. In a week's time a mixture of crystals formed. The larger ones were carefully removed by tweezers and analysed.

|                    | Found. | Calculated for<br>$\text{La}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ . |
|--------------------|--------|---------------------------------------------------------------------------------------------------------|
| Lanthanum oxide .. | 27.24  | 27.69                                                                                                   |
| Potassium oxide .. | 8.47   | 8.03                                                                                                    |

This would indicate the formation of the alum. The amount obtained, 0.1868 grm., was used in the analysis. The remaining smaller crystals and some of the alums gave, on analysis, 47.37 per cent lanthanum oxide. This indicates the coincident formation of the hexahydrate of Frerichs and Smith, which carries 49.17 per cent lanthanum oxide (*Liebig's Ann. Chem.*, xcxi., 460).

In hopes of repeating the above in another experiment with more dilute solutions the current was allowed to run for twenty-three hours. The crystals, a mixture, gave lanthanum oxide, 46.2 per cent, and potassium oxide, 9.71 per cent.

A third experiment was carried out on a larger scale, using 150 c.c. of the double lanthanum potassium sulphate solution and 75 c.c. of the lanthanum sulphate. The current, 0.1 ampère, was allowed to run for forty eight hours. No crystallisation took place, but four fractions were obtained in the concentration. None of them gave results on analysis worthy of mention.

Similar experiments were carried out with sodium sulphate and the current continued for fifty hours. The solutions obtained were evaporated under reduced pressure. The analysis of the crystals gave:—

|                    | Found. | Calculated for<br>$\text{La}_2(\text{SO}_4)_3 \cdot 2\text{Na}_2\text{SO}_4 \cdot 18\text{H}_2\text{O}$ . |
|--------------------|--------|-----------------------------------------------------------------------------------------------------------|
| Lanthanum oxide .. | 27.40  | 27.41                                                                                                     |
| Sodium oxide .. .. | 9.78   | 10.47                                                                                                     |

This is doubtless a mixture and its study could not be of interest.

Another experiment with sodium sulphate was carried out with a larger current, 0.4 ampère, and continued for seventy hours. Some pretty needles separated contaminated with fine crystals. It was quite impossible to remove the former and free them from the mother-liquor—they were so soluble. The small crystals on analysis proved to be Cleve's double sulphate (*Ber.*, xi., 912).

|                    | Found. | Calculated for<br>$\text{La}_2(\text{SO}_4)_3 \cdot 2\text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O}$ . |
|--------------------|--------|------------------------------------------------------------------------------------------------------|
| Lanthanum oxide .. | 36.30  | 36.04                                                                                                |
| Sodium oxide .. .. | 13.15  | 13.87                                                                                                |

Experiments were carried out with rubidium sulphate. It may be remarked that it mattered not which one of the alkaline sulphates was used in the mixture; any effort to concentrate by heating always caused a precipitation of the hydrated sulphate, some double salt, or both. In a solution made of rubidium and lanthanum sulphates in theoretical amounts and evaporated in a vacuum desiccator, thin, flat silky crystals formed which, on analysis, gave:—

|                                                                                         | Lanthanum oxide. | Rubidium oxide. |
|-----------------------------------------------------------------------------------------|------------------|-----------------|
| Calculated for—                                                                         |                  |                 |
| $\text{La}_2(\text{SO}_4)_3 \cdot \text{Rb}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ .. | 25.77            | 14.78           |
| $3\text{La}_2(\text{SO}_4)_3 \cdot 2\text{Rb}_2\text{SO}_4$ .. ..                       | 43.62            | 16.77           |
| Found .. .. .                                                                           | 43.10            | 17.64           |

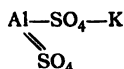
\* Presented in abstract at the Cleveland Meeting of the American Chemical Society. From the *Journal of the American Chemical Society*, 1904, xxvi., No. 1.

Lanthanum sulphate is, as remarked, much more soluble in cold than in hot water, the solubility, according to Mosander, being 1 part in 115 of water at 100° and 1 in 6 parts at 13° (*Journ. Prakt. Chem.*, xxx., 380). As one of us (M.) was forced to withdraw from the laboratory, an effort was made in closing up the work to concentrate the rubidium solution at the temperature 45–50° C. The vessel was placed in an air-bath, the temperature regulated, and the evaporation facilitated by drawing a current of dry air over the liquid by means of a suction-pump. Crystals were obtained giving a percentage of 40.4 of lanthanum oxide which would indicate the formation  $\text{La}_2(\text{SO}_4)_3 \cdot \text{Rb}_2\text{SO}_4$ , where the percentage of lanthanum oxide is 39.13. The body belongs to the type  $\text{Di}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4$  (Marignac, *Liebig's Ann. Chem.*, lxxxviii., 242) and  $\text{Di}_2(\text{SO}_4)_3 \cdot \text{Ti}_2\text{SO}_4$  (Zschiesche, *Journ. Prakt. Chem.*, cvii., 98).

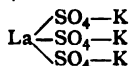
Efforts were also made to prepare an ammonium alum, but with no more satisfactory results.

Perhaps under more suitable conditions, as spontaneous evaporation at low temperatures, the lanthan-alums may be prepared. Apparently, however, the property possessed by lanthanum sulphate of forming a stable compound with 9 molecules of water with slight elevations of temperature interferes materially with the formation of the alum. Muthmann and Rölig have observed its formation at zero (*loc. cit.*).

Attention has been directed by F. W. Clarke to the alum type—



and the suggestion made that lanthanum, which readily gives  $\text{La}_2(\text{SO}_4)_3 \cdot 3\text{K}_2\text{SO}_4$ , forms the model—



These specific properties of lanthanum render the formation of the alum more difficult.

# ELEVENTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED DURING 1903.\*

By F. W. CLARKE.

(Concluded from p. 164).

## Fluorine.

JULIUS MEYER (*Zeit. Anorg. Chem.*, xxxix., 313) has re-determined the atomic weight of fluorine by a new and simple method. Calcium oxide was converted into fluoride, with suitable precautions, and from the ratio thus measured the atomic weight of fluorine was easily computed. First, the oxide, slaked with water, was converted by hydrochloric acid into chloride. The latter was then repeatedly evaporated with pure hydrofluoric acid, and the resulting fluoride was ignited to constant weight at a red heat. The data, with vacuum weights, are as follows:—

| Weight CaO. | Weight CaF <sub>2</sub> . | Atomic weight F. |
|-------------|---------------------------|------------------|
| 6.1883      | 8.6215                    | 19.036           |
| 4.2736      | 5.9548                    | 19.042           |
| 6.2931      | 8.7658                    | 19.029           |
| 5.7767      | 8.0485                    | 19.038           |
| 4.9836      | 6.9426                    | 19.033           |

Mean . . . 19.036

Calculated by Meyer with O = 16 and Ca = 40.136.

## Iron.

A new determination of the atomic weight of iron, by Baxter (*Proc. Am. Acad.*, xxxix., 245), is based upon the

\* From the *Journal of the American Chemical Society*, xxvi., No. 3.

analysis of ferrous bromide. This salt was sublimed in porcelain tubes, dissolved in acidulated water, oxidised by potassium dichromate, and then mixed with a solution of silver nitrate. The silver bromide was collected on a Gooch filter and weighed. In two experiments the ferrous bromide was also balanced against silver alone. The data, as published, involve a number of corrections, which complicate the table of results. The following corrected weights have kindly been supplied to me by Dr. Baxter. They are reduced to a vacuum, and the calculations represent O = 16, Ag = 107.93, and Br = 79.955.

| Weight FeBr <sub>2</sub> . | Weight AgBr. | Weight Ag. | Atomic weight Fe. |
|----------------------------|--------------|------------|-------------------|
| 3.55929                    | 6.19873      | —          | 55.856            |
| 3.07448                    | 5.35450      | —          | 55.852            |
| 2.96102                    | 5.15696      | —          | 55.849            |
| 4.00791                    | 6.97983      | —          | 55.862            |
| 2.96102                    | —            | 2.96234    | 55.854            |
| 4.00791                    | —            | 4.00937    | 55.871            |

Mean . . . 55.857

One correction still remains to be applied. Ferrous bromide, like the corresponding bromides of cobalt and nickel, when sublimed in porcelain, becomes contaminated with small quantities of sodium bromide. In this case the contamination amounted to 0.138 per cent. Corrected for this impurity the final value for Fe becomes 55.871. This agrees fairly well with the value 55.883 previously determined by Richards and Baxter. The number 55.88 is probably quite nearly correct.

As a check upon the accuracy of this work, Dr. Baxter also re-determined the ratio between silver and bromine. In three independent experiments his data are as follows:—

| Weight Ag. | Weight AgBr. | Ratio Ag : AgBr. |
|------------|--------------|------------------|
| 4.77783    | 8.31754      | 57.4428          |
| 5.87977    | 10.23533     | 57.4459          |
| 4.82995    | 8.40809      | 57.4441          |

Mean . . . 57.4443

This is nearly identical with the mean derived from Stas' experiments, viz., 57.4445. From the two experiments of the ferrous bromide series in which both Ag and AgBr were determined, the values for the ratio became 57.444 and 57.442.

## Antimony.

The electrolytic method for determining the atomic weight of this element has been investigated by Cohen and Strengers (*Proc. Amsterdam Acad.*, v., II., 543). Antimony and silver were thrown down simultaneously in the same circuit, the former from solutions of trichloride. It was found that the atomic weight thus obtained increased with the concentration of the antimony solution, varying from 120.78 to 121.92, when Ag = 107.93. The concentrations varied from 3.3 to 83.3 grms. of chloride in 100 grms. of solution, and the gain in apparent atomic weight was quite regular. The method therefore involves unknown uncertainties, and is not applicable to the purpose of measuring the atomic weight in question.

## Lanthanum.

In the report of this committee for 1902 a set of determinations, by Jones, of the atomic weight of lanthanum was given, and also certain criticisms of the work by Brauner, who claimed that it was vitiated by constant errors. In his reply to Brauner, Jones (*Zeit. Anorg. Chem.*, xxxvi., 92) claims to show that the alleged errors do not exist, and new data are given in corroboration of the former series. In the new work hygroscopic impurity was carefully guarded against, and the lanthanum sulphate was proved to be neutral. Five syntheses of sulphate from oxide, conducted in porcelain crucibles, gave the subjoined results, when O = 16 and S = 32.06.



| Weight $\text{La}_2\text{O}_3$ . | Weight $\text{La}_2(\text{SO}_4)_3$ . | Atomic weight La. |
|----------------------------------|---------------------------------------|-------------------|
| 1'2161                           | 2'1132                                | 138'79            |
| 1'6311                           | 2'8342                                | 138'81            |
| 1'7804                           | 3'0938                                | 138'79            |
| 1'4168                           | 2'4619                                | 138'80            |
| 1'9702                           | 3'4235                                | 138'80            |
| Mean .. ..                       |                                       | 138'80            |

The value found in 1902 was  $\text{La} = 138'77$ .

In these experiments the oxide used was perfectly white. Brauner and Pavlicek heated their oxide, not in porcelain, but in platinum crucibles, and found a higher value for the atomic weight, 139'04. Jones now gives the results of two experiments in platinum, and obtains similar values, as follows:—

| Weight $\text{La}_2\text{O}_3$ . | Weight $\text{La}_2(\text{SO}_4)_3$ . | Atomic weight La. |
|----------------------------------|---------------------------------------|-------------------|
| 1'2820                           | 2'2264                                | 139'02            |
| 1'3885                           | 2'4110                                | 139'07            |

In this case the oxide was somewhat discoloured, and most so near the wall of the platinum crucible. Jones regards this as due to a slight oxidation of the material, which would account for the higher atomic weight. The controversy, however, is probably not yet ended. Determinations of atomic weight by some process radically different from either the sulphate or the oxalate method may be necessary to settle the questions at issue.

#### Cerium.

Upon the atomic weight of cerium two important and elaborate memoirs have appeared; one by Brauner and Batek (*Zeit. Anorg. Chem.*, xxxiv., 103), the other by Brauner alone (*Zeit. Anorg. Chem.*, xxxiv., 207). Only the results of the two investigations can be given here; for details the original publications must be consulted.

First, the salt  $\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$  was dehydrated by heating to  $440^\circ$ . The anhydrous sulphate was then reduced by ignition, with suitable precautions, to  $\text{Ce}_2\text{O}_3$ . In this way, operating upon various fractions of material, Brauner and Batek found:—

| Weight sulphate. | Weight oxide. | Atomic weight Ce. |
|------------------|---------------|-------------------|
| 1'5074           | 0'9130        | 140'12            |
| 1'7979           | 1'08945       | 140'32            |
| 1'5937           | 0'9665        | 140'13            |
| 2'6240           | 1'5895        | 140'18            |
| 1'2161           | 0'7370        | 140'38            |
| 1'4974           | 0'9130        | 140'12            |
| 1'2192           | 0'7386        | 140'21            |
| Mean .. ..       |               | 140'21            |

Calculated with  $\text{O} = 16$ ,  $\text{S} = 32'06$ , and reduction to a vacuum.

In the second paper, Brauner gives a series of experiments in which the salt  $\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$  was directly reduced to  $\text{Ce}_2\text{O}_3$ . The data, with vacuum weights throughout, are as follows:—

| Weight octahydrate. | Weight oxide. | Atomic weight Ce. |
|---------------------|---------------|-------------------|
| 1'98989             | 0'96175       | 140'26            |
| 1'99154             | 0'96251       | 140'25            |
| 2'33919             | 1'13027       | 140'17 (?)        |
| 1'95882             | 0'94679       | 140'28            |
| 1'20961             | 0'58453       | 140'21            |
| 1'54162             | 0'74504       | 140'24            |
| 1'67748             | 0'81074       | 140'25            |
| 2'02736             | 0'97985       | 140'26            |
| Mean .. ..          |               | 140'24            |

Rejecting the third of these values, in which Brauner suspects a possible loss during the experiment, the mean of the remaining seven becomes 140'25.

For the determinations which the authors carried out by

the oxalate method, the data are too complex for full citation here. Only results can be given. The method involves two steps; first, ignition of cerium oxalate to  $\text{Ce}_2\text{O}_3$ ; and secondly, determination of  $\text{Ce}_2\text{O}_3$  in another portion of oxalate by titration with potassium permanganate. From the ratio between  $\text{Ce}_2\text{O}_3$  and  $\text{Ce}_2\text{O}_4$  the atomic weight of cerium is calculated. By this process Brauner and Batek made the following eighteen determinations:—

|         |    |    |         |
|---------|----|----|---------|
| 140'28  |    |    | 140'075 |
| 140'465 |    |    | 140'075 |
| 140'45  |    |    | 140'20  |
| 140'165 |    |    | 140'10  |
| 140'29  |    |    | 140'32  |
| 140'43  |    |    | 140'30  |
| 140'38  |    |    | 140'37  |
| 140'075 |    |    | 140'42  |
| 140'33  |    |    | 140'05  |
| Mean    | .. | .. | 140'265 |

One more experiment by this method, cited by Brauner in the second paper, gave  $\text{Ce} = 140'246$ . As the final result of both investigations, Brauner gives—

$$\text{Ce} = 140'25$$

This agrees with earlier work by Brauner, and also with determinations by Robinson, and should replace all other measurements of this atomic weight.

#### Radium.

In the report of this committee for 1902, Madame Curie's determinations of the atomic weight of radium were cited. Her data have since appeared in full, as follows (*Ann. Chim. Phys.*, [7], xxx., 140):—

| Weight $\text{RaCl}_2$ . | Weight $\text{AgCl}$ . | Atomic weight Ra. |
|--------------------------|------------------------|-------------------|
| 0'09192                  | 0'08890                | 225'3             |
| 0'08936                  | 0'08627                | 225'8             |
| 0'08839                  | 0'08589                | 224'0             |

Calculated with  $\text{Ag} = 107'8$  (?) and  $\text{Cl} = 35'4$ . Madame Curie regards the value  $\text{Ra} = 225$  as correct to within about one unit. This conclusion finds confirmation in the work of W. Marshall Watts (*Phil. Mag.*, [6], v., 203; and, for Radium, vi., 64), who has studied relations between the spectra of the elements and the squares of their atomic weights. By comparing the spectra of Ba and Ra he finds  $\text{Ra} = 225'05$ ; and for the spectra of Hg and Ra,  $\text{Ra} = 224'63$ . Mean of all his observations,  $\text{Ra} = 224'89$ . Runge and Precht, on the other hand, comparing the spectra of Ca, Sr, Ba, and Ra, concluded that the atomic weight of radium is 257'8 (*Physikal. Zeit.*, iv., 285). The apparent instability of radium offers an argument in favour of this higher value. In a later paper Runge (*Phil. Mag.*, [6], vi., 698) criticises Watts's method of calculation, and argues that it is entirely fallacious.

#### Miscellaneous Notes.

**Krypton.**—A new determination of the density of krypton, by Ramsay, gives the value 40'81 (*CHEMICAL NEWS*, lxxxvii., 159). The atomic weight therefore is 80'62.

**Bismuth.**—Adie, seeking to determine the atomic weight of bismuth, obtained discordant results, which were found to be due to small quantities of silica in the material studied (*Proc. Cambridge Phil. Soc.*, xii., 240). To this impurity he ascribes the discrepancies between the measurements of previous observers. A preliminary experiment upon pure bismuth gave  $\text{Bi} = 208'8$  approximately.

**Tellurium.**—The atomic weight of tellurium has been discussed by Koethner (*Zeit. Anorg. Chem.*, xxxiv., 403), by Seubert (*cit. Anorg. Chem.*, xxxv., 205), and by Pellini (*Gazz. Chim. Ital.*, xxxiii., II., 35). Koethner, from an examination of all the published determinations, concludes that the most probable value is  $\text{Te} = 126'71$ , when  $\text{H} = 1$ .

Seubert argues in favour of  $\text{Te} = 126.6$  or  $127.6$  with  $\text{O} = 16$ . Pellini, considering the anomalous position of tellurium in the periodic system, suggests a possible impurity in the form of a radio-active element of higher atomic weight.

**Numerical Relations.**—This subject is discussed by Mills, under the caption "Numerics of the Elements" (*Phil. Mag.*, [6], v., 543). An attempt is made to compute the atomic weights from a general mathematical formula, and arguments are adduced to show that the most probable value for  $\text{O}$  is  $15.94$  when  $\text{H} = 1$ . Another paper, by Hughes (*CHEMICAL NEWS*, lxxxviii., 298), develops relations which suggest that the elements may be constituted analogously to the organic radicals. Still a third paper, by Henry Wilde, also deserves attention (*Mem. Manchester Lit. Phil. Soc.*, xlviii., 1).

**International Atomic Weights.**—The report of the smaller International Committee for 1903 (*Journ. Am. Chem. Soc.*, 1903, xxv.) has been sharply criticised by Ostwald (*Zeit. Phys. Chem.*, xlii., 637). To this criticism, which related mainly to the standard of values, Seubert published a reply (*Zeit. Anorg. Chem.*, xxxv., 45). The subject of a standard has also been discussed by Winkler (*Chem. Zeit.*, xxvii., 918), whose paper called forth a rejoinder by Landolt and Ostwald (*Ber.*, xxxvi., 3759). In a still later note, Winkler answers his critics (*Ber.*, xxxvii., 4299). For the purposes of this report, these references to literature are sufficient.

## PROCEEDINGS OF SOCIETIES.

### CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, March 16th, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MESSRS. L. F. GUTTMANN, J. H. POLLOK, F. D'O. MEARS, and M. Blood were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Harold S. Hammond, Government Laboratory, Kingston, Jamaica; Laurel Cecil F. Oldfield, Lincoln College, Oxford; William D. Page, Constitutional Club, London, W.C.; Thomas L. D. Porter, B.Sc., 161, Downsell Road, Stratford, E.; Louis John E. Riley, Port-of-Spain, Trinidad; Henry Heron Smith, 76, Plumstead Common Road, Plumstead.

Of the following papers, those marked \* were read:—

\*46. "Mercuric Nitrite and its Decomposition by Heat." By PRAFULLA CHANDRA RAY.

Mercuric nitrite, hitherto unknown, can be obtained by evaporating *in vacuo* over sulphuric acid, the solution left by decomposing mercuric chloride with silver nitrite. It occurs in groups of light yellow needles which slowly deliquesce and decompose when exposed to the air. The salt and its aqueous solution are stable when sealed up out of contact with air. The solid salt is largely decomposed by water. When heated at about  $100^\circ$  in a vacuum, it melts and intumesces, decomposing for the most part into mercurous nitrate and nitric oxide, but also, to a small extent, into red, crystalline mercuric oxide and the gases of decomposed nitrous anhydride. Mercurous nitrate is also the main product of the decomposition of mercurous nitrite at about  $200^\circ$ , but in this case the nitrate occurs principally as a pseudo-sublimate, derived from the vapours of the decomposing nitrite (*Proc.*, 1903, xix., 78; *Trans.*, 1903, lxxxiii., 491).

### DISCUSSION.

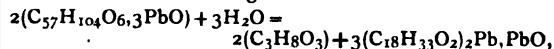
Dr. DIVERS said that, in order to understand the production of mercurous nitrate and nitric oxide from mercuric nitrite, it was necessary (1) to bear in mind that nitric peroxide acts freely on metallic silver to form nitrate and

nitric oxide, and has no action whatever on silver nitrite, and that silver nitrate heated with nitric oxide, even at temperatures well below  $200^\circ$ , interacts with it to form nitrite and nitric peroxide; (2) to assume the same to be true of mercurous nitrite and nitrate; and (3) to refer to the decomposition by heat of mercurous and silver nitrites. Silver nitrite, heated only moderately, behaves almost like mercuric nitrite, except that half the silver is left in the metallic state. But when it is decomposed rapidly, the silver nitrate produced is small in quantity, most of the silver being left in the metallic state and much nitric peroxide escaping. When mercurous nitrite is heated, the ultimate products are also mercurous nitrate and nitric oxide, together with about half of the metal in the free state. But here, visibly, the first products are, for the most part at least, metal and nitric peroxide, which then, at some distance off, interact to form mercurous nitrate and nitric oxide. It can, therefore, hardly be doubted that the products of decomposition of two molecules of mercuric nitrite by heat are represented by  $2\text{Hg} + 4\text{NO}_2$ , which then become  $(\text{Hg} \cdot \text{O})_2 + 2\text{NO}$ , without much loss by vaporisation at the relatively low temperature at which the salt decomposes.

\*47. "Note on the Higher Glycerides." By JAMES BALLANTYNE HANNAY.

The higher glycerides, as represented by purified stearin, olive oil, linseed oil, castor oil, cotton-seed oil, rape oil, and earth-nut oil, are all capable of entering into direct combination with lead oxide, the new compounds being formed by heating the oils with excess of finely divided litharge at  $170^\circ$  to  $180^\circ$ , and dissolving out the product with chloroform, petroleum, or carbon tetrachloride. The substance obtained resembles wax, and has no sharp melting-point; it begins to soften at  $120^\circ$ , becomes viscous at  $150^\circ$  to  $160^\circ$ , and is quite limpid at  $190^\circ$ . It commences to boil and decompose at about  $280^\circ$ , the temperature varying a few degrees according to the oil used.

In the case of the olein derivative, the composition may be represented by the formula  $\text{C}_3\text{H}_5\text{O}_3 \cdot \text{Pb}_3(\text{C}_{18}\text{H}_{33}\text{O}_2)_3$ , where the three atoms of lead are seen to replace six atoms of hydrogen, three in molecules of glycerol, and three in those of oleic acid. This compound probably represents the first step in saponification by lead oxide. The oleic acid derivative, when dissolved in ether, is decomposed by cold water in the following manner:—



glycerol and a basic lead oleate being thereby produced. Lead glyceryl oleate and the corresponding stearate, linoleate, and ricinoleate resemble mercury thymyl acetate in forming a double compound with lead acetate.

The foregoing compounds are stable and not affected by fractional precipitation, neither is the lead displaced by metallic sodium, phosphoric oxide, or sulphur trioxide. The free linkings of the unsaturated glycerides were unaffected by the combination with lead oxide, for the iodine number remained unchanged.

The combination of the lead glyceryl oleate with sulphur chloride is similar to the products obtained from the unsaturated glycerides in general. An examination of the action of sulphur chloride on oils showed that with due precaution the sulphur chloride additive compound was identical in type with the substances obtained by the addition of bromine, iodine, and oxygen. The sulphur chloride saturated the free linking, thus behaving as a bivalent radicle. The oxygen absorption was determined for the same oils, and the results of all these combinations have been tabulated.

During these investigations, no evidence has been obtained of the existence of Hazura's linolenic or *iso*-linolenic acid with six free linkings (with eighteen in the triglyceride); all the iodine numbers are well under the limit for pure linoleic acid with four free linkings, this deficiency being due to the presence of oleic or other acid of a more saturated type.

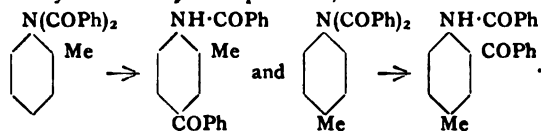
Experiments were made with numerous samples from which the acid had been obtained by three different methods, but only linoleic acid with four linkings was obtained, this result agreeing with that of Reformatsky, who could find no evidence of the existence of acids with six free linkings.

The author showed that Hazura's method of separation with alkaline permanganate has a very variable effect, the oxidation proceeding further than the formation of hydroxy-acids. Hence it is concluded that no acid with an iodine number higher than that corresponding with four free linkings exists in natural oil.

\*48. "Isomeric change of Diacylanilides into Acylaminoketones. Transformation of the Dibenzoyltoluidines into the Isomeric Benzoylaminomethylbenzophenones." By FREDERICK DANIEL CHATTAWAY and WILLIAM HENRY LEWIS.

It has been shown recently that acyl groups must be included among those which, under suitable conditions, can pass from the nitrogen of an aromatic amine into the nucleus, displacing a hydrogen atom in a para- or ortho-position relatively to the nitrogen.

An excellent illustration of this intramolecular re-arrangement is furnished by the behaviour of the dibenzoyltoluidines, which at a high temperature under the influence of hydrogen chloride are converted into the isomeric benzoylaminomethylbenzophenones, thus:—



In the transformation of dibenzoyl-*o*-toluidine, when the acyl group might take up a para- or an ortho-position relatively to the nitrogen, the former is assumed, apparently exclusively. In the transformation of dibenzoyl-*p*-toluidine, an ortho-position only is available.

As in the case of the analogous transformation of the acylchloroamines, interchange between the *p*-hydrogen atom and the acyl group takes place more readily than that in which the hydrogens in the ortho-positions are concerned.

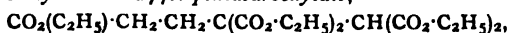
The yield of 4-amino-5-methylbenzophenone is about 50 per cent, and that of 2-amino-5-methylbenzophenone about 20 per cent calculated on the weight of the corresponding toluidine used.

These hitherto unknown aminoketones were described together with a number of their more important derivatives.

\*49. "The Action of Ethyl  $\beta$ -Iodopropionate on Ethyl Disodioethanetetracarboxylate." By OSWALD SILBERRAD.

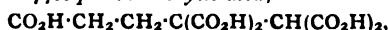
The investigation of the interaction between ethyl  $\beta$ -iodopropionate and ethyl disodioethanetetracarboxylate has led to the discovery of the following new compounds:—

Ethyl butane- $\alpha\gamma\delta\delta$ -pentacarboxylate,—



boils at 215–218° under 17 m.m. pressure, and yields haloid derivatives which readily give rise to a lactic acid.

Butane- $\alpha\gamma\delta\delta$ -pentacarboxylic acid,—



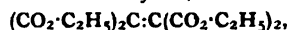
readily loses carbon dioxide, forming butane- $\alpha\gamma\delta$ -tricarboxylic acid, which melts at 122°, and not at 116–120° as previously given (Auvers, Kibner, and Megenbarg, *Ber.*, 1891, xxiv., 2895).

Ethyl  $\Delta^7$ -dihydrouconate ( $\Delta^8$ -butylenedicarboxylate),  $\text{CO}_2(\text{C}_2\text{H}_5) \cdot \text{CH}_2 \cdot \text{CH} : \text{CH} \cdot \text{CH}_2 \cdot \text{CO}_2 \cdot \text{C}_2\text{H}_5$ , boils at 120–125° under 17 m.m. pressure. The formation of this substance, which constitutes one of the minor fractions, is probably due to the condensation of two molecules of ethyl  $\beta$ -iodopropionate with loss of hydriodic acid.

Hexane- $\alpha\gamma\gamma\delta\delta$ -hexacarboxylic acid was obtained by the saponification of the corresponding ester, which, however,

was not isolated in a pure condition; the constitution of the acid was established by its conversion, with loss of carbon dioxide, into hexane- $\alpha\gamma\delta\delta$ -tetracarboxylic acid, which latter appears to be identical with Sell and Easterfield's  $\alpha\alpha$ -diglutaric acid.

Ethyl ethylenetetracarboxylate,—



is also produced in small quantities, and was identified by conversion into its acid potassium salt. Its formation is probably due to the action of iodine, produced by the decomposition of ethyl  $\beta$ -iodopropionate, on ethyl disodioethanetetracarboxylate.

\*50. "The Heat of Formation of Glucinum Chloride." By JAMES HOLMES POLLOK.

This research was undertaken in order to obtain the heat of formation of glucinum chloride direct from the metal, for this constant is required in calculating the heats of formation of the other salts of glucinum from the heats of neutralisation of the various acids by the base glucina.

A sample of ground beryl from Limoges was employed in the preparation of pure glucina, and this oxide was converted into the anhydrous chloride by heating with carbon (from cane-sugar) in a current of dry chlorine, the metal being finally obtained from the salt by the action of metallic sodium in a nickel crucible.

On dissolving the metal in hydrochloric acid contained in a calorimeter, it was found that the heat of formation of glucinum chloride in solution was 199.5 calories. The heat of solution of the anhydrous chloride in water is 44.5 calories, and deducting this from the foregoing number, the heat of formation of dry anhydrous glucinum chloride is found to be 155 calories.

\*51. "Note on the Composition of Distilled Oil of Limes and a New Sesquiterpene." By HERBERT EDWARD BURGESS and THEODORE HENRY PAGE.

The authors have isolated and identified *l*-terpineol (m. p. 35°) as forming a large proportion of the oxygenated constituents of the oil. The peculiar odour of the oil which is attached to the terpeneol fraction is due to an isomeric liquid terpeneol of slightly lower boiling-point. A new sesquiterpene of partially olefinic nature was also identified, which boils at 131°/9 m.m. and at 262–263° (uncorr.)/756 m.m., in the latter case, with slight decomposition; it has a sp. gr. 0.873 at 15°, and is optically inactive;  $n_D$  is 1.4935 at 15°, and 1.4910 at 19.5°. The sesquiterpene, for which the name "*limene*" is proposed, was characterised by the formation of a trihydrochloride (m. p. 79–80°); this was the only well-defined derivative obtained, and from it the hydrocarbon can be readily regenerated. The same sesquiterpene has been identified in hand-pressed lime oil and lemon oil, and the other oils of this series are being examined for it.

52. "The Nature of a Solution of Iodine in Aqueous Potassium Iodide." By CHARLES HUTCHINS BURGESS and DAVID LEONARD CHAPMAN.

Jakowkin (*Zeit. Physikal. Chem.*, 1894, xiii., 529; 1896, xx., 14) has shown that the distribution of iodine between carbon disulphide and an aqueous solution of potassium iodide of varying concentration accords with the assumption of a dissociable compound  $\text{KI}_3$ . Dawson's observations (*Trans.*, 1901, lxxix., 238), conducted at a different temperature, confirm this conclusion. The work of the authors has been directed towards the measurement of the

relative velocities of the  $\text{I}^-$  and  $\text{I}_3^-$  ions by two independent methods; firstly, by a careful comparison of the iodine carried over to the anode by the same current in cells containing respectively aqueous potassium iodide and a solution of iodine in aqueous potassium iodide, and, secondly, by a comparison of the conductivities of the same solutions.

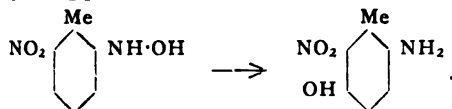
The mean values of the relative velocities of the  $\text{I}_3^-$  and  $\text{I}^-$  ions, determined by the first and second methods, are 0.556 and 0.553 respectively. These practically identical results,

obtained by two different processes, are regarded as confirming the conclusions of previous observers.

A new and symmetrical method of writing the equilibrium equations of the foregoing solution was suggested.

53. "The Reduction of 2:6-Dinitrotoluene with Hydrogen Sulphide." By JULIUS BEREND COHEN and JOSEPH MARSHALL.

In the course of an experiment in which 2:6-dinitrotoluene was reduced with hydrogen sulphide in alcoholic ammonia, a quantity of 2-nitro-4-amino-*m*-cresol was obtained together with 6-nitro-*o*-toluidine. The nitroaminocresol crystallises in dark orange needles, which dissolve readily in dilute acids and alkalis and melt at 190° with decomposition; it forms colourless salts with the mineral acids, and is precipitated from its solution in alkalis by carbon dioxide. On oxidation with lead peroxide and dilute sulphuric acid, it yields 2-nitrotolquinone, which crystallises in ruby-red prisms melting at 64–65°, and is converted into 2-nitrotolquinol by reduction with sulphur dioxide; the latter crystallises in scarlet needles melting at 117–118°, and dissolving in aqueous alkalis to a violet solution. A solution of bleaching powder in the presence of hydrochloric acid converts nitroaminocresol into chloronitrotolquinone, which crystallises in pale yellow, spear-shaped crystals melting at 70–71°. The nitroaminocresol is formed by the incomplete reduction of 2:6-dinitrotoluene to 6-nitro-*o*-tolylhydroxylamine in the same way that trinitrobenzene and trinitrotoluene are converted into the hydroxylamino-compounds (Cohen and Dakin, *Trans.*, 1902, lxxxii., 26). The hydroxylamine derivative, on boiling with dilute hydrochloric acid, changes into the corresponding *p*-aminocresol:—



54. "Acid Esters of Methylsuccinic Acid." By WILLIAM ARTHUR BONE, JOHN JOSEPH SUDBOROUGH, and CHARLES HENRY GRAHAM SPRANKLING.

The methyl hydrogen salts of succinic, *cis*- and *trans*-dimethylsuccinic, and tetramethylsuccinic acids have been obtained by the addition of methyl alcohol to the corresponding anhydrides; they melt respectively at 58°, 38°, 49°, and 63°.

The methyl hydrogen salts of the unsymmetrical monomethylsuccinic, *gem*-dimethylsuccinic, and trimethylsuccinic acids have been prepared by three distinct methods:—(a) from the anhydride, (b) from the neutral ester, and (c) from the acid. The oily products obtained by all three methods from methylsuccinic acid, give practically the same dissociation and esterification of constants.

From *gem*-dimethylsuccinic acid, two distinct acid methyl esters have been obtained; one of these melts at 52° and has a lower dissociation, and also a lower esterification constant than its isomeride, which melts at 40.5°.

The methyl hydrogen salts obtained from trimethylsuccinic acid are oils, the esters obtained from the anhydride and from the acid appear to be identical, but differ from the oil obtained from the neutral ester as regards their dissociation and esterification constants. The results obtained indicate that there is no direct relationship between the two sets of constants.

The esterification constant tends to decrease with an increase in the number of methyl groups present. Walker's generalisation with respect to the dissociation constants does not hold good, as in the succinic series the ratio—

$$\frac{K \text{ acid}}{K \text{ acid ester}}$$

increases from 2.12 for succinic acid to 27.0 for tetramethylsuccinic acid. The value for *a* in Wegscheider's equation,  $aK = K_1 + K_2$ , gradually decreases from 0.945 for succinic acid to 0.074 for tetramethylsuccinic acid,

55. "A Note on Phenyltrimethylallylammonium Compounds." By ALFRED WILLIAM HARVEY.

Before the publication of a paper by H. O. Jones (*Trans.*, 1903, lxxxiii., 1400), the author had attempted to resolve phenyltrimethylallylammonium compounds into active constituents. His results, although differing in important details, confirm the conclusions deduced by Barrowcliff and Kipping (*Trans.*, 1903, lxxxiii., 1141) and by Jones (*loc. cit.*).

56. "Estimation of Hydrogen Peroxide in the presence of Potassium Persulphate, by means of Potassium Permanganate." By JOHN ALBERT NEWTON FRIEND.

A correct estimate of the amount of hydrogen peroxide present in a mixture of that substance and potassium persulphate solution is not given under ordinary conditions by titration with potassium permanganate, the values obtained being always too low.

It is found that the proportion of permanganate required to decompose the hydrogen peroxide is subject to the following variations:—(1) It increases with the speed of titration, (2) it varies inversely with the concentration of the persulphate and the volume of the titrated solution, (3) it increases and finally reaches the theoretical amount when the concentration of the sulphuric acid is increased.

A fairly accurate estimation may therefore be obtained if the time of titration is short, and the volume titrated is small, whilst the concentration of the sulphuric acid is fairly great.

57. "A Comparison of the Products of the Hydrolysis of Potato Starch with those obtained from Cereal Starches." By JAMES O'SULLIVAN.

In this investigation, it was established by six series of hydrolytic experiments on potato, Lintner's malt, barley, maize, and rice starches, with both malt-extract and diastase, that the products of the hydrolysis of potato starch, as regards the percentages of maltose and dextrin, bear no quantitative relationship to those yielded by the other starches, and that therefore the products of the hydrolysis of the other starches could not be inferred from the hydrolysis of potato starch.

## PHYSICAL SOCIETY.

Ordinary Meeting, March 25th, 1904.

Dr. R. T. GLAZEBROOK, F.R.S., President, in the Chair.

A PAPER entitled "Note on the Measurement of Small Inductances and Capacities and on a Standard of Small Inductance" was read by Prof. FLEMING.

The author referred to a paper read before the Society last year by himself and Mr. W. C. Clinton, in which a motor-driven commutator was employed to measure small inductances. It had since been found that very good results could be obtained in the measurement of small inductances by Prof. Anderson's method, by using a telephone in place of a galvanometer and a buzzer in the battery-circuit. The author had found that for long solenoids, at least 50 diameters long, the inductance could be calculated with an accuracy of about 1 per cent by the rule:—Inductance in c.m. = (Length of wire in one unit length of solenoid) × (Total length of wire in the whole solenoid in c.m.). Numerous measurements were given showing the agreement between the measured and predicted inductance of various solenoids and the limits of the dimension-ratio within which the rule was valid. It was also shown that by means of such a predetermined inductance small capacities, such as those of Leyden jars, could be easily measured. A standard of inductance graduated in microhenries suitable for electric resonance experiments and Hertzian wave telegraphy was exhibited. The standard consisted of a spiral of copper wire with special means for producing gradual variation or definite variation in the inductance thrown into a circuit.

Mr. A. CAMPBELL said they had lately measured some self-inductances of the order of 100 microhenries (100,000 c.m.) at the National Physical Laboratory. The method used was practically that of Max Wien as modified by Dolezalek. In the original method two contiguous arms of a Wheatstone's bridge consist of (1) the inductive coil (under test) shunted by a known non-inductive resistance, and (2) an unknown (preferably variable) inductance with an adjustable resistance in series. The other two arms are non-inductive resistances, with a slide-wire between them. The indicating instrument (either an optical telephone or a vibration-galvanometer) is tuned to respond only to a particular frequency, and the source of current is worked at this frequency. Thus, although the source may give a quite irregular wave-form, the instrument practically ignores all but one sine-curve component, and the formula can therefore be easily obtained. The product and the quotient of the inductances in the network are given in terms of non-inductive resistances and  $p$ , where  $p = 2\pi n$ ,  $n$  being the frequency of the current-source. From these equations the inductances can be separately determined. The modification described by Dolezalek consists in using a current-source giving a nearly pure sinoidal wave and an ordinary telephone as an indicating instrument. The current-generator is a "microphone hummer." In this a telephone-plate (P) carries a microphone (M). A current from two accumulators is passed through M and the primary of an induction-coil, the secondary being connected with a polarised magnet acting on P. Dolezalek states that, by the use of a "hummer" and a variable inductance standard, inductances of about 0.1 microhenry (100 c.m.) can be readily measured to within 1 or 2 per cent. For obtaining frequencies above 1000  $\sim$  per second, Mr. Campbell has found it desirable to replace the telephone-plate by a steel bar 2.5 c.m. in diameter. In order to make the note in the detecting telephone pure, the latter is tuned by means of a small screw pressing against the ferrotype plate.

Mr. W. DUDDELL expressed his interest in the paper, and asked what precautions were taken to ensure that the resistances and connections used were free from self-induction and capacity. He thought that some of the variations in the figures given in Table I. of the paper might be due to these effects.

Dr. FLEMING, in reply, said that in connection with all measurements of small inductances it was essential to continually check the measurements by the aid of a small known inductance made as he described. We were all apt to forget that the ideal "bridges" drawn on paper were not identical with real "bridges," in that the latter had small inductance and capacity in all the arms and connections, and hence formulæ obtained on the assumption that certain branches were absolutely inductionless and non-capacious were not strictly correct. In some experiments he had found that great errors were introduced merely by using ordinary flexible twin wires for connections, as these had quite a sensible capacity per metre. In all methods employing the telephone as a detector we were testing the observer quite as much as the method, and not only a perfectly quiet room, but a very acute ear, were necessary if good results were to be obtained. It was becoming important to possess methods of measuring accurately inductances of less than 1 microhenry. The author sketched a plan which he said he had not yet put into practice, but which seemed promising. It involved the use of a Hertz rectangle, and the measurements were made to depend upon the identity in time-period between two conducting paths connecting the same points.

"A Hot-Wire Ammeter for Measuring very small Alternating Currents" was exhibited and described by Prof. FLEMING.

The author said that in alternating-current work, particularly in taking the power-factor of small transformers and of short lengths of cables, the need had been felt for an ammeter not involving the use of iron capable

of measuring currents as small as 0.01 or less of an ampère. He exhibited an ammeter capable of being made to read currents as small as 0.002 with fair accuracy. The arrangement was as follows:—Two very fine platinoid or constantin wires, about 1 metre long and 0.05 or even 0.02 m.m. diameter, are supported on a wooden rod with arrangements for adjusting their tensions. These wires are 5 m.m. apart, and are held down at the centre by delicate spiral springs. The two wires are embraced at the middle by a small loop of paper carrying a very small plane mirror. These wires are enclosed in a box, the lid of which carries a lens. By this means the light of a straight carbon filament of a glow-lamp, or of a slit illuminated by an arc-lamp reflected by this small mirror, can be focussed on a screen of ground glass. If a current is passed through one of these wires it sags down slightly, and the square root of the displacement of the image on the screen is almost exactly proportional to the current passing. The instrument can be quickly calibrated by applying a known voltage to the ends of the wire, the resistance having previously been measured corresponding to the range of current used. Examples were given of the use of such an instrument. Suitable very fine wires of pure metals and alloys are now prepared by Messrs. Hartmann & Braun of Frankfurt. These fine wires require "ageing" before use by intermitting a current through them for 12 hours or more.

Mr. W. DUDDELL expressed his interest in the paper and said it was difficult to make a satisfactory instrument. The author had said that with a fine constantin wire it was possible to measure currents as small as 2 milliamperes. He asked if this was calculated or observed. There should be a standard method of comparing these instruments, and he suggested using the current necessary to give a deflection equal to one-quarter of the scale distance. Mr. Duddell pointed out that many dynamometers were more sensitive than the instrument shown, and asked the author the order of magnitude of the currents which it was necessary to measure in aerial conductors.

Dr. W. WATSON exhibited and described a form of ammeter for small alternating currents. The current to be measured flows through a piece of iron wire bent into the form of a right angle. This is linked with a similarly shaped piece of nickel wire forming part of a galvanometer circuit. The thermo-E.M.F. at the junction, produced by the heating effect of the current, sends a current through the galvanometer which can be measured in the usual way. The current to be measured is practically proportional to the deflection of the galvanometer.

Mr. A. CAMPBELL described a form of ammeter by Rubens; and Mr. W. A. PRICE referred to an instrument similar to that shown by the author, in which the sag of the wire is measured by the movement of an aluminium pointer.

Dr. FLEMING said he was quite aware that electro-dynamometers had been designed for measuring very small alternating currents, but they were more difficult to make and calibrate than the simple form of hot wire instrument he now exhibited. He had no doubt that the use of a constantin wire of 0.02 m.m. diameter would greatly increase the sensitiveness of the instrument, though he had not actually tried it for want of time. As regards Mr. Duddell's question about the magnitude of the currents flowing into a Wireless Telegraph aerial, that of course depended upon the size of the aerial. In the case of an ordinary 180 foot single aerial the average value was about 0.5 to 1 ampère, but that depended of course on the frequency of the groups of oscillations. The author said that although in the ammeter he exhibited the sag of the working wire was indicated by a mirror, yet he had also used an index-needle, and he had no doubt a good commercial instrument might be made on this plan.

A paper on "Energy of Secondary Röntgen Radiation" was read by Mr. C. G. BARKLA.

To measure the intensity of radiation electroscopes

were placed in a primary beam of Rontgen rays and in a secondary beam proceeding from air in a direction perpendicular to that of propagation of the primary rays. By comparison of the two rates of leak when no absorbing plates were used and when similar aluminium plates were placed before each electroscope, it was found that the absorptivity of the secondary rays differed from that of the primary by less than 5 per cent of its value. It was, however, found that a secondary beam of the same intensity as the primary would produce a slightly different number of ions in a given volume of air, consequently the radiations differ slightly in character. The difference in what may be called the "ionising powers" was evidently greater when the primary beam consisted of more penetrating rays. The fraction of energy lost in secondary radiation was very nearly, if not entirely, independent of the character of the primary radiation. The law which the author had previously found to govern the intensity of radiation from gases, was found to be equally applicable to those light solids which are the source of a radiation differing little in character from the primary, *i.e.*, the energy of secondary radiation from these substances situated in a beam of definite intensity is proportional to the quantity of matter through which the primary beam passes. In the passage of X-radiation through air under normal atmospheric conditions, the diminution of intensity due to secondary radiation is of the order of magnitude 0.02 per cent per centimetre. This is a large fraction of the total loss of energy for penetrating rays passing through air. Applying experimental results to J. J. Thomson's calculation of the loss of energy per c.m. due to the passage through a medium containing ions, and considering the electrons as the source of radiation, the number of these per c.c. of air under normal conditions is of the order of  $10^{14}$ . By using the secondary radiation from copper as the primary, approximately the same fraction of the energy was lost in secondary radiation; hence we have quantitative results showing that this radiation from copper, though differing in character from the primary radiation producing it, is of the same nature. Evidence was also found pointing to the conclusion that even from metals which are the source of a secondary radiation differing greatly from the primary, the energy of secondary radiation is of the order of magnitude given by gases and light solids of the same mass.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 10, March 7, 1904.

**Europium.**—G. Urbain and H. Lacombe.—The authors find that the magnesium salts of europium have nearly the same solubility as those of bismuth, whilst the same salt of gadolinium is considerably more soluble. Hence it is tolerably easy to separate europium from gadolinium in the tail of the fractionation. The authors then proceed to calculate the atomic weight of europium.—(1) By the transformation of the hydrated sulphate into the anhydrous sulphate; (2) by the transformation of the anhydrous sulphate into the oxide; and (3) by the transformation of the hydrated sulphate into the oxide. The results reciprocally check one another, and give almost identical results. The authors therefore conclude that the atomic weight of europium is definitely 151.79, and they estimate that this number differs from the real number by less than 0.06.

**Action of Carbonic Anhydride on the Ammonium Metals.**—Etienne Rengade.—Carbonic anhydride reacts on sodium-ammonium and potassium-ammonium. Below  $-50^{\circ}$  it forms exclusively the alkaline carbonate, with

evolution of hydrogen. At a rather higher temperature an alkaline formate is simultaneously produced, a portion of the hydrogen evolved in the first reaction being thus absorbed. This production of formate by nascent hydrogen and carbonic anhydride in presence of an ammonium metal is analogous to the synthesis effected by M. Moissan by means of the alkaline anhydrides and carbonic anhydride.

**General Method of Preparation of Anhydrous Chlorides.**—C. Matignon and F. Bourion.—A mixture of chlorine and sulphur chloride constitutes an excellent chlorinating agent for oxides. The reaction must take place at a low temperature, and it allows of the easy preparation of anhydrous chlorides, even in the case of the most exothermic oxides.

**Phenylurethanes of Sugar.**—L. Maquenne and W. Goodwin.—It is known that phenylisocyanate reacts on polyatomic alcohols belonging to the mannite group, as well as on saccharines, to give insoluble phenylurethanes. The authors apply the same reaction to the reducing sugars and to polyoses without previous hydrolysis. The sugar with a slight excess of carbanile is diluted with two or three times its volume of anhydrous pyridine and then brought to boiling-point. The reaction is rapid, and, after a quick drying process, a crystalline salt separates out. By this method, hitherto unknown phenylurethanes are prepared, as those of the pentoses, hexoses, and certain hydrolysable polyoses, such as lactose, trehalose, and melezitose. Their composition only varies between very narrow limits; however, the analyses are in all cases in perfect accordance with the theoretical composition.

**Allyl- and Propenyl-alcoyl Ketones.**—E. E. Blaise.—The author examines the reaction by which allyl ketones are transformed into propenyl ketones. He finds that the condensation of nitriles with allyl iodide in presence of zinc really gives only the allyl ketone, the propenyl ketone being formed on the elevation of temperature.

**Compounds of Saccharose with certain Metallic Salts.**—D. Gauthier.—A compound of saccharose with sodium iodide has already been prepared and described. Whilst endeavouring to reproduce this compound by the same method, the author obtained a crystalline product in very distinct prisms. He proves the formula of this substance to be  $C_{12}H_{22}O_{11}, NaI, 2H_2O$ , differing from that found by Gill. However, this formula is analogous to those proved for compounds of saccharose with potassium iodide and lithium iodide:— $C_{12}H_{22}O_{11}, KI, 2H_2O$ ;  $C_{12}H_{22}O_{11}, LiI, 2H_2O$ . The metallic sulphocyanides play parts analogous to those of chlorides, bromides, and iodides, and the author succeeds in obtaining definite products with some of these and saccharose. Ammonium, potassium, and sodium sulphocyanides give beautiful prismatic crystals, whose composition is represented by the formulae  $C_{12}H_{22}O_{11}, NH_4CNS, 1.5H_2O$ ,  $C_{12}H_{22}O_{11}, KCNS, H_2O$ ,  $C_{12}H_{22}O_{11}, NaCNS, H_2O$ . Barium sulphocyanide also gives a compound showing prismatic crystals and having the formula  $C_{12}H_{22}O_{11}, Ba(CNS)_2, 2H_2O$ .

## MISCELLANEOUS.

**Royal Institution.**—On Tuesday next, April 12, at 5 o'clock, Mr. L. C. Miall delivers the first of three lectures at the Royal Institution on "The Transformations of Animals"; on Thursday, April 14th, at the same hour, Professor Dewar commences a course of three lectures on "Dissociation"; and on Saturday, April 16, at three o'clock, Mr. Cyril Davenport delivers a lecture on "Mezzotints," to be followed on the two succeeding Saturdays by lectures on (1) "Comeos," (2) "Jewellery." The Friday Evening Discourse on April 15 will be delivered by Count Vay De Vaya, on "Korea and the Koreans"; on April 22 by Colonel David Bruce on

"Sleeping Sickness in Uganda"; and on April 29 by Dean Robinson, on "Westminster Abbey in the Early Part of the 17th Century."

**Adulterated Drugs and Chemicals.**—The United States Department of Agriculture has recently issued a Bulletin, No. 80, on the adulteration of drugs and chemicals. The Bulletin contains three articles; the first is on inferior drugs and insidious methods of deception; the second is on rose geranium oil and its substitutes; and the third on phenacetin, methods of analysis and commercial status. There is a continual cry for cheaper drugs, and in the effort to meet this demand, and at the same time to make a profit, adulteration has spread. This pamphlet will be useful in pointing out the principal methods of adulteration practised, though many tricks, such as colouring with harmless vegetable dyes, are without any danger; still, even in such a case, the fact that the goods are dyed ought to be stated. The article on phenacetin is of some importance; it deals with the history, methods of manufacture, physical and chemical tests, and other points connected with this useful drug. Numerous chemical tests have been proposed for the detection of acetanilid in phenacetin, but so far the ideal method has not been found; among the best of those in use we may mention the bromine test, the saponification test, the mercurous nitrate test, the iodophenol test, and lastly, the isonitrite reaction. Many conflicting statements have been made as to the reliability of the isonitrite test for acetanilid, and though it is at present recognised as an identifying test by the British, German, and United States Pharmacopœias, neither of the two former mentions it in connection with phenacetin, and the latter does not recognise this chemical. We may take it, therefore, that this test has failed.

**The Decomposition of Carbonic Oxide in the Blast-furnace.**—Rudolf Schenk and P. Zimmermann.—The reversible reaction  $2\text{CO} \rightleftharpoons \text{C} + \text{CO}_2$  has been the subject of a great many papers. It may be considered from the point of view of the variation of volume, or the variation of pressure which results. The authors have made use of a special apparatus suitable for this purpose, and they have examined the influence of finely divided metals and oxides held in pumice stone. It results, according to them, that the oxides of iron, nickel, and cobalt do not cause any decomposition of the CO, but, on the other hand, the finely divided metals favour it considerably. The examination of the phenomena of the dissociation of CO at different temperatures in the presence of nickel and cobalt, show that the reaction at a high temperature ( $445^\circ$ ) is bimolecular,  $2\text{CO} = \text{C} + \text{CO}_2$ , while at a low temperature ( $310^\circ$ ) it is the result of two reactions:—(1)  $\text{CO} = \text{C} + \text{O}$ , (2)  $\text{CO} + \text{O} = \text{CO}_2$ . In the presence of finely divided iron the reaction is more complex.—*Berichte*, vol. xxxvi., p. 1231.

## MEETINGS FOR THE WEEK.

- MONDAY, 11th.**—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "Volatilisation of Lead Oxide from Lead Glazes into the Atmosphere of a China Glast Sagger and its Effect upon the Leadless Glaze Ware in the same Sagger" and "The Preparation of Lead Glazes of Low Solubility, and some Points to be observed in the Process," by W. A. Thomason. "Action of certain Solutions upon Aluminium and Zinc," by Watson Smith.
- TUESDAY, 12th.**—Royal Institution, 5. "The Transformations of Animals," by Prof. L. C. Miall, F.R.S.
- WEDNESDAY, 13th.**—Faraday Society, 8. "Alloys of Copper and Arsenic" by Arthur H. Hiorne. "A New Primary Battery" and "Note on Determining Accurately the Percentage of Ozone in Gases not Dissociated by Moderate Heat," by E. G. P. Bousfield.
- Society of Public Analysts, 8. "The Microscopic Examination of Metals" (Illustrated by Lantern Slides), by J. H. B. Jenkins and D. G. Riddick. "Cod Liver Oil and other Fish Oils" and "Note on Mushroom Ketchup," by J. F. Liversidge, F.I.C.

- THURSDAY, 14th.**—Royal Institution, 5. "Dissociation," by Prof. Dewar, F.R.S., &c.
- FRIDAY, 15th.**—Royal Institution, 9. "Korea and the Koreans," by The Rt. Rev. Monsignor the Count Vay de Vaya and Lusko, D.P.R.N., K.C.J.C.
- SATURDAY, 16th.**—Royal Institution, 3. "Mezzotints," by Cyril Davenport, F.S.A.

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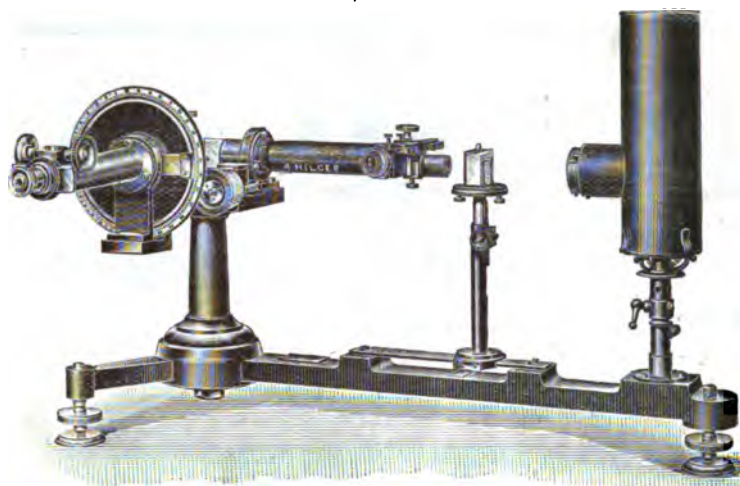
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## CONTENTS.

| ARTICLES:—                                                                                               | PAGE |
|----------------------------------------------------------------------------------------------------------|------|
| The Liquid State, by P. J. Beveridge, M.A., B.Sc.                                                        | 181  |
| The Separation of Chromium and Vanadium, by Paul Nicolardot                                              | 182  |
| The Separation of Iron and Chromium by means of Fused Potassium Nitrate, by F. Southerden, B.Sc., F.I.C. | 183  |
| Notes on Rapid Soil Analysis, by Vincent Edwards, F.C.S., F.I.C.                                         | 183  |
| Contributions to the Chemistry of the Rare Earths, by Chas. Baskerville and Hazel Holland                | 184  |
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S.                              | 186  |
| PROCEEDINGS OF SOCIETIES—                                                                                |      |
| CHEMICAL SOCIETY                                                                                         | 188  |
| NOTICES OF BOOKS                                                                                         | 190  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                    | 191  |
| MISCELLANEOUS                                                                                            | 192  |
| MEETINGS FOR THE WEEK                                                                                    | 192  |

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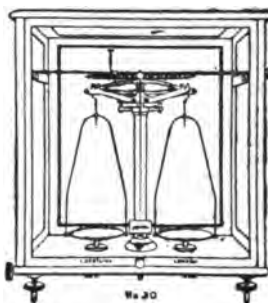
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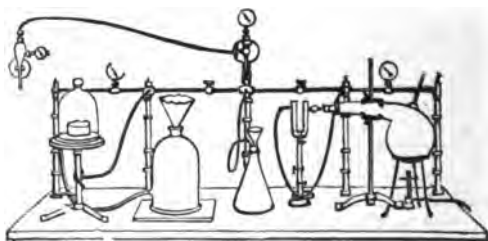
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2316.

## THE LIQUID STATE.

By P. J. BEVERIDGE, M.A., B.Sc.

(Concluded from p 169).

We turn next to Avogadro's law. This has long been accepted as having a certain limited and rather indefinite bearing on solution. Nernst, in the preface to his work on Theoretical Chemistry, places it alongside of the doctrine of energy as the most important foundation of all chemical investigations:—"The rule of Avogadro, which seems to be an almost inexhaustible horn of plenty for the molecular theory" (English translation, p. xiii., and *vide* p. 30). And so, on turning to the case of osmotic pressure, whereby the dissolved substance exercises osmotically the same pressure as it would exercise if it were a gas under the influence of Avogadro's law, we find the same qualified acceptance of the law as governing solutes. Well, if we assume this application of Avogadro's law, and if we assume also that the osmotic pressure of the solute is due to the kinetic translatory energy of its molecules within the liquid, then osmotic pressure becomes perfectly intelligible.

For suppose we have the usual apparatus in illustration of osmosis, a porous cell so treated as to make it "semi-permeable," permeable that is to the molecules of solvent, but not to the molecules of solute. Then the molecules of solvent, being free to pass, will produce no effective pressure, while the pressure arising from the motion of the molecules of the solute will be filtered off, so to speak, from all other pressures and attractions. The molecules of the solute will strike against the semi-permeable wall and rebound, they will "bombard" the wall as gas molecules bombard their containing vessel, and if we assume that their translatory kinetic energy is equal to the energy they would have as gas molecules, and also that Avogadro's law holds good with reference to them, then their osmotic pressure will be identical with their calculated gas pressure, and that is experimentally found to be the case.

But everything seems to show that we must go further than this. If solutes are themselves liquids we must apply Avogadro's law to the molecules of the solvent as well. We must apply it to liquids in general. It must be stated that the mean volumes occupied by all the molecules in any particular solution however complicated are the same. We are indeed compelled by thermodynamics to admit that their mean kinetic energy is the same; let us admit that their molecular volume is also the same. Indeed, it does not seem very clear how the one could be true without the other.

Let us see whether this will at all simplify matters. Let us apply it to the law of the diminution of vapour pressure in solutions already quoted. Ostwald points out that no satisfactory explanation has been given of this very simple law. It can be shown by thermodynamics to be a necessary consequence of osmotic pressure, but such thermodynamical reasoning consists in a *reductio ad absurdum* of the opposite proposition, and therefore gives no real explanation at all.

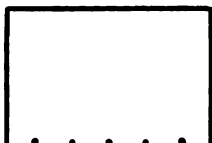


FIG. A.

Let A be the surface of a volatile liquid capable of containing, say, 5 liquid molecules, and enclosed above. A concrete number is taken for simplicity. Within some

certain time  $t$ , 5 molecules moving faster than their neighbours will have escaped from the attraction of the liquid, and will take the gaseous form. In another space of time  $t$  5 more will escape, but there will also be re-bounds, bringing some of the escaped molecules back into the attraction of the liquid. A point will be reached at length by gradual concentration of the vapour when on an average in the time  $t$  5 molecules leave the liquid and 5 are absorbed by it again. This gives the maximum vapour pressure at the particular temperature. Suppose we denote this pressure by 5.

But now suppose, say, 2 of these molecules of volatile liquid are suddenly replaced by non-volatile molecules, molecules therefore which from whatever reason are unable to leave the liquid. According to Avogadro's law they will fill the same space as those they replace. (They will not necessarily fill the same space as was occupied before, for there have been great pressure changes within the line of surface tension; but they fill the same space as each of the remaining molecules of volatile solvent now fill). See Fig. B. So in the time  $t$  there will be 3 molecules escaping

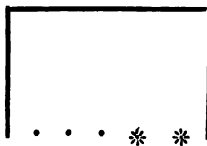


FIG. B.

from the liquid; at first 5 will be absorbed, but as only 3 escape the vapour pressure will be gradually lowered until the point is reached when 3 molecules are striking the liquid in the time  $t$ , and 3 escaping; then the pressure will become constant again. Consequently, the pressure which we denoted by 5 will be reduced to a pressure which we must denote by 3. And thus the reduction of the whole pressure is proportional to the number of the molecules exchanged for molecules of non-volatile solute. This is the law already quoted. And so a clear and simple explanation of the law of the reduction of pressure in solution comes in view. Conversely the argument can be used with equal cogency to prove that the molecules occupy the same space in accordance with Avogadro's law.

Further consideration will show that there is here an underlying assumption that the heat required to separate a molecule of vapour from the pure liquid and from the solution is the same; that, in fact, the same kinetic energy of an escaping molecule will enable it to overcome the attraction of the liquid in either case. This is not quite correct, for these heats differ by the heat of dilution of the solution (the heat appearing when a grm.-molecule of pure solvent is added to the solution); but in dilute solutions this may be neglected.

Yet a further step. We cannot confine our conclusions to the molecules within one liquid. It must be asserted, as a consequence, that the kinetic translatory energy of every molecule of all liquids is the same at the same temperature, and likewise that, given the volume of the grm.-molecule of any liquid at a certain temperature, we can calculate therefrom what the kinetic pressure must be. The total pressure is a different thing; it is dependent upon inter-molecular attraction.

It may be advisable to note that to hold that the kinetic translatory energy of all liquid molecules at the same temperature is the same and is equal to the gas energy, does not, *primâ facie* at least, involve the extension of Avogadro's law. Of the two, the statement with regard to the kinetic energy rests perhaps on surer ground.

Some years ago I was able to come across a remarkable confirmation of this statement. It occurred to me that if we were dealing with liquids of monatomic molecule, so that the whole of their heat energy is translatory and none of it intra-molecular, then, by the solidification of the liquid, the whole of this translatory motion must be stilled. Thus, if it is true that the energy of this motion is equal to

the gaseous molecular kinetic energy at the same temperature, it follows that it will be equal to  $3f$ . Now experiments on the vapour pressure of amalgams have shown that metallic molecules are probably monatomic, and this is confirmed by vapour density observations on mercury, cadmium, and zinc. Accordingly, I argued that in liquefying a metallic molecule heat must be supplied equal to  $3f$ ;  $3f$  will be the molecular heat of liquefaction. There are disturbing considerations to be taken into account, incipient polymerisation, the changes of volume, &c., but notwithstanding this I found an even closer correspondence to theory than I had expected, and recent determinations of vaporisation heats have brought it closer still. The degree of correspondence with theory is comparable with the results obtained by Ramsay by vapour pressure of amalgams for the molecular weights of the metals, and the delicacy of manipulation renders the accurate determination of these liquefaction heats at high temperatures an exceedingly difficult operation. The law I have already stated as follows:—

*The molecular liquefaction heat of a liquid whose molecules are monatomic is equal to the translatory kinetic energy of a gas molecule at the temperature of liquefaction; that is, it is equal to  $3f$ .*

This law I published in the *CHEMICAL NEWS* in 1897 (vol. lxxvi., p. 264), and attention was again drawn to it by Mr. H. Crompton at last year's meeting of the British Association. I append the table I first used in illustration of the law, amended by the employment of some of Mr. Crompton's heats of liquefaction, which—though he does not give a reference—appear to have been more accurately determined since the first publication of the law. The newer data strengthen the case.

| Melting-point.<br>Abs. t. | Heat of liquefaction (unit mass). |                 |  | Ratio.<br>A : B. |
|---------------------------|-----------------------------------|-----------------|--|------------------|
|                           | A.<br>Calculated.                 | B.<br>Observed. |  |                  |
| Silver .. 1227°           | 33·72                             | 26·7            |  | 1·27             |
| Cadmium .. 588°           | 15·52                             | 13·66           |  | 1·14             |
| Mercury .. 233°           | 3·45                              | 2·82            |  | 1·22             |
| Zinc .. 685°              | 31·01                             | 28·1            |  | 1·10             |
| Platinum .. 2048°         | 31·24                             | 27·2            |  | 1·15             |
| Lead .. 598°              | 8·67                              | 6·4             |  | 1·35             |
| Palladium .. 1773°        | 49·6                              | 36·2            |  | 1·37             |
| Copper .. 1355°           | 64·2                              | 49·1            |  | 1·31             |
| Thallium .. 562°          | 8·18                              | 7·2             |  | 1·14             |
| Aluminium .. 927°         | 103                               | 80              |  | 1·29             |
| Gold .. 1330°             | 20·2                              | 16·3            |  | 1·24             |
| Sodium .. 365°            | 47·48                             | 32·7            |  | 1·45             |
| Potassium .. 335°         | 25·6                              | 15·7            |  | 1·63             |
| Tin .. 503°               | 12·63                             | 13·62           |  | 0·93             |
| Bismuth .. 533°           | 7·6                               | 12·64           |  | 0·603            |
| Gallium .. 286°           | 12·85                             | 19·11           |  | 0·673            |

The case of tin, bismuth, and gallium is peculiar, but the first two have non-metallic tendencies, and their non-metallic relatives are allotropic. Perhaps sodium and potassium await more accurate determination.

The calculation utterly fails when we come to deal with binary molecules, such as  $\text{Br}_2$ ,  $\text{I}_2$ , and this is itself a corroboration of the law. But one would hardly hesitate to predict that the new gases, argon, krypton, and xenon, will be found to conform with it, unless indeed the close proximity of the solid to the gaseous state in the first two—in the first especially—be found to interfere. Their heats of liquefaction per unit mass would be 6·4, 3·83, 3·12. They may fall 20 per cent below this, but in the case of these permanent gases, as with the most volatile metals, the agreement will probably be much closer. The liquefaction points of the other new gases of the atmosphere are not determined.

Considerations like these open up a wide ground for research. One or two directions may be indicated. We may assume provisionally that in the case of liquids dissolved in volatile liquids the law of variation of vapour pressures is accurately given by the equation quoted, and

refer the divergences noted with increasing concentration to heat of dilution alone, according to the remarks above, and carefully observing these divergences we may collate them with changes in the heat of dilution at varied concentration, and in the surface tension and in the mean molecular volume, all of which must be intimately connected. Again, there is great need for a more extensive and accurate determination of liquefaction heats. And so we may conclude appropriately with the words of Pattison Muir:—"What is wanted now is, therefore, not only further experimental determinations of physical constants of chemically similar compounds, but a great development of the general theory of the structure of matter, especially in the direction of applying this theory to liquid and solid bodies" (*Watts' Dictionary*, Article "Atomic and Molecular Weights").

## SEPARATION OF CHROMIUM AND VANADIUM.

By PAUL NICOLARDOT.

THE separation of chromium and vanadium is very difficult because these elements are found together in considerable quantity, and in almost equal proportions. The separation is almost impossible by the use of ammonium sulphohydrate, and very laborious by the aid of ammonia, as shown by M. Carnot. The method proposed by M. L. Hôte is also not easy of application, and that devised by MM. Matignon and Bourion, in which the action of sulphur is substituted for that of carbon, has the disadvantage of being very complicated.

*Separation in the State of Chlorochromic Acid.*—It appears to me that the separation of chromium and vanadium would be very simple if the chromium were in the state of chlorochromic acid. It is therefore necessary to treat a mixture of chlorides, chromates, and anhydrous alkaline vanadates with sulphuric acid containing in solution a little anhydride in a very dry bulb. At first the heat evolved is sufficient to start the reaction without artificial heating. The evolution of chromyl chloride is, however, assisted by attaching the apparatus to an air-pump and creating a vacuum, and heating with a spirit-lamp towards the end of the reaction. The last vapours are driven off by a current of dry air. On account of the energy of the reaction a little vanadium is lost mechanically. In order to retain this the bulb should be attached to a little wash-bottle containing a small quantity of strong sulphuric acid. The sulphuric acid used in this reaction should contain a small quantity of anhydride in order to avoid the partial decomposition of the chromyl chloride by the water which is produced during the reaction.

*Details of the Experiment.*—The compound containing the chromium and the vanadium is melted with a mixture of chlorate and carbonate ( $4\text{ClO}_3, \text{CO}_3\text{Na}_2$ ), or with a large excess of chlorate alone. The small quantities of iron and manganese, if present, are separated by the ordinary methods. The alkaline salts are then concentrated, dried, and melted. The molten mass is completely washed until there is an absence of colour in the wash-water. If there is no iron present, the mixture can be directly melted in the little bulb which was used for the first reaction. Attached to the bulb a little wash-bottle is placed containing a little concentrated sulphuric acid, then a flask containing a dilute solution of soda, through which the gases evolved during the reaction can bubble. The apparatus is attached to a pump.

A current of dry air traverses the apparatus, and a few drops of strong sulphuric acid containing a little anhydride are introduced through a dropping funnel. The reaction at once starts. When it becomes less brisk the pump is brought into play, and a few more drops of acid added, and the air or dry  $\text{HCl}$  is allowed to enter bubble by bubble. Then the bulb is slightly heated and finally washed until it no longer evolves red vapours. In a very few minutes the whole reaction ceases. The bulb must not be over-



heated, a temperature of 60° being usually sufficient. We prove that all the chromium has been extracted by treating the contents of the bulb on a water-bath, first with  $\text{SO}_3\text{Na}_2$ , then with excess of ammonia. The precipitate of  $\text{Cr}_2\text{O}_3$ , if it is abundant, still retains some vanadium, and the process must be repeated. The vanadium can be separated at first if the operation has worked satisfactorily. To estimate it, a little alcohol is added to the liquor. This is heated on a water-bath until all the alcohol is driven off, and then titrated with permanganate. The presence of molybdenum does not interfere with this volumetric estimation.

**Separation by means of Condensed Ferric Oxide.**—If the compound, which contains vanadium and chromium, also contains a large quantity of iron, the chromium and vanadium can be separated in a still simpler manner. The compound is attacked by hydrochloric acid, the solution then oxidised by nitric acid, or better still by chloric acid, and after this it is evaporated on the water-bath in presence of an excess of hydrochloric acid. I have shown that under these conditions the greater quantity of the acid is driven out (about two-thirds), and a precipitate of condensed ferric oxide is formed which retains the metalloids, the metals remaining in solution.

Chromium sesquioxide, if it contains less than two molecules of water when heated, is always partially transformed into chromic acid. Therefore the chromium in the solution is transformed into chromic acid. It then can be separated, all except the traces which remained in the precipitate of condensed iron oxide; these remaining traces of chromium can be extracted from the ferruginous deposit by treating it with hot water containing some drops of dilute alcohol. After boiling the solution which holds in suspension the precipitate detached from the capsule, ammonium sulphate is added. All the chromium goes into solution, whilst the vanadium remains entirely in the ferric precipitate, from which it can be separated by first washing with ammonia and then fusing with alkaline salts to separate the last traces. The vanadium is then estimated volumetrically.

These two methods can also be used to separate chromium from other metalloids and other metals.—*Comptes Rendus*, March 28, 1904.

## THE SEPARATION OF IRON AND CHROMIUM BY MEANS OF FUSED POTASSIUM NITRATE.

By F. SOUTHERDEN, B.Sc., F.I.C.

THE method to be described contains no new principle, depending merely on the oxidation of the chromium by means of  $\text{KNO}_3$  and  $\text{KHSO}_4$ . The use of fused  $\text{KHSO}_4$  in analysis is of ancient origin, though less appreciated for ordinary qualitative work than it deserves to be, and it has long been employed in conjunction with  $\text{KNO}_3$  for the estimation of Cr in chrome iron ore. But it does not seem to have been observed before that this fusion and oxidation can be carried out in a few minutes in a test-tube, under proper conditions, without injury to the tube. Apart from ease of manipulation, the substitution of a test-tube for the generally recommended platinum foil or charcoal block may be considered an advantage in dealing with students.

Instead of fusing with  $\text{KHSO}_4$  and then adding  $\text{KNO}_3$  and raising the temperature as in many operations, the  $\text{KNO}_3$  is used as the flux and the  $\text{KHSO}_4$  added in small quantity as the decomposing agent. This brings about a much more vigorous oxidation, and gives a more readily fusible "melt."

The precipitate containing the hydroxides of iron and chromium is smeared over a small basin and rotated over the Bunsen flame until approximately dry. It is then scraped into a dry test-tube, one or two smallish crystals of  $\text{KNO}_3$  added, and the whole melted by cautiously heating over a low flame. Then a little piece of anhydrous  $\text{KHSO}_4$  is dropped in, and the heating continued until brown fumes are copiously evolved. The tube is cooled, water added,

and  $\text{NH}_4\text{OH}$  until alkaline. The fused mass readily dissolves, leaving the iron (and any aluminium) behind as oxide or hydroxide. The residue is filtered off and dissolved in a little concentrated  $\text{HCl}$ , and tested in the usual way for iron with  $\text{K}_4\text{FeCy}_6$ . To the filtrate  $\text{AgNO}_3$  is added, which gives a red precipitate of  $\text{Ag}_2\text{CrO}_4$ . A few drops of dilute acetic acid may be necessary to give the chromate indication if any large excess of ammonia is present.

The whole series of operations may be carried out in a few minutes, starting with the wet precipitate. A very insoluble sample of the green oxide of chromium was found to be oxidised almost instantaneously to chromate by this method, and the insoluble violet,  $\text{CrCl}_3$ , reacts at once with the fused  $\text{KNO}_3$  without addition of  $\text{KHSO}_4$ . Even chrome iron ore is sufficiently decomposed to give a decided indication of chromium in the aqueous extract.

The Technical College, Finsbury.

## NOTES ON RAPID SOIL ANALYSIS.

By VINCENT EDWARDS, F.C.S., F.I.C.

ANALYTICAL chemists employed in manure works are often called on to examine and report on soils, also to suggest treatment with fertilisers, an occupation which tends to increase, and one which shows how neglected is this investigation, which should be of supreme importance. Works chemists have difficulties to contend with, they are often short of time, the sample supplied is too small, and so on; in fact, a works chemist bears the same relationship to his official brother as the merchant does to the navy seaman, both doing useful work but under different conditions. Having given some attention to this matter I venture to make some suggestions as to the methods to be employed when the above conditions prevail, as well as a few remarks on the various items.

The preparation of the sample is comparatively simple, breaking up if necessary, and passing through a  $\frac{1}{4}$ -inch sieve, which removes stones, the mineralogical character of which should be noted, then grinding in an agate mortar.

**Water.**—Five grms. is a good quantity to take, drying for five hours in the water-oven. Organic matter and loss on ignition: 5 grms. ignited gently for five hours over an argand lamp, then for half-an-hour over a Bunsen with a rose.

**Phosphoric Acid.**—10 grms. are gently ignited, evaporated to dryness on water-bath with 20 c.c.  $\text{HCl}$ , taken up with 10 c.c. more acid, and the insoluble matter filtered off. On cooling, nitric acid solution of ammonium molybdate is added to the filtrate, the beaker allowed to stand forty-eight hours, when the yellow precipitate is collected, washed with small quantities of water till free from acid, dissolved in hot dilute ammonia, the solution evaporated to dryness, and weighed in a platinum dish; the residue contains 3.5 per cent  $\text{P}_2\text{O}_5$ .

**Iron and Alumina.**—In this rapid analysis these can be estimated together. The ignited soil is treated with 20 c.c.  $\text{HCl}$ , evaporated to hard dryness, taken up with hot water and acid, the silicates (insoluble in  $\text{HCl}$ ) dried, ignited, and weighed. The iron and alumina in the filtrate precipitated with ammonia; this repeated so as not to bring down any lime, dried, ignited, and weighed; the  $\text{P}_2\text{O}_5$  already found being deducted. **Lime.**—Solid ammonium oxalate in powder is added to filtrate, boiled for some time, allowed to stand overnight, and the precipitate collected, then either ignited or the amount found by the volumetric method with permanganate as given in Sutton. **Potash** is best estimated by Tatlock's method, taking 10 grms., treating with 10 c.c.  $\text{HCl}$ , evaporating to dryness, filtering, evaporating filtrate to dryness, dissolving with hot water and  $\text{HCl}$ , filtering off any insoluble, evaporating slowly with excess of  $\text{PtCl}_4$ ; a very excellent method.

**Nitrogen.**—A Kjeldahl determination with 10 or even 20



grms.; a special copper flask made by Messrs. Griffin, as suggested by me some years ago, can be used. It may be here pointed out how very necessary a "blank" is; the best  $\text{H}_2\text{SO}_4$  sometimes contains an appreciable quantity of nitrogen. Nitrates may be looked for by some special colour method.

With regard to the various items:—

**Water.**—Some text-books say this is of no value. Is this certain? A constant succession of very wet samples of soil show either abnormal rainfall or bad system of drainage. Very wet land will be almost certain to benefit by active manures.

**Organic Matter and Loss on Ignition.**—There can be no doubt about the importance of a moderate amount of organic matter. If the amount is very low, suggestions might be made as to addition of farmyard manure. In tropical lands a complete exhaustion of the organic matter of the soil is a serious affair, and may make all the difference between a valuable and worthless plantation. Tennant pointed out what a calamity the total loss of vegetable mould was in Ceylon. It is a question if the aid of the Kjeldahl process could not be invoked to estimate rapidly by colour the organic matter in the soil.

**Phosphoric Acid.**—This determination is of prime importance; nothing can be expected from a soil denuded of this ingredient, as too many are. It is wonderful to think how neglected is this key to the true value of a soil. It is not too much to say that the admirable method suggested by Dr. Dyer, whose important work has justly earned the gratitude of all chemists, at once throws a vivid light on this essential matter. Nitrogen is to some extent supplied by the atmosphere; potash pretty freely present in most soils, even if in rather an inert state; but phosphoric acid, so much depends on the intelligence of the agriculturalist for its sufficient supply. A chemist is asked what percentage of phosphoric acid in the soil calls for treatment. I would say that when under 0.25 per cent on the dry soil enrichers might be applied; under 0.10 per cent the application of suitable phosphatic manure essential. The same figures might also apply to nitrogen and potash; in fact, a scale of valuation of land might be founded on this 0.25 per cent, and be useful to those farmers, auctioneers, &c., who would find a whole figure more satisfactory than decimals.

**Iron and Alumina.**—A moderate percentage of these soluble in HCl is a decided advantage, and their amount, large or small, give the chemist some assistance in judging as to what form the proposed manure should take if iron is only present in small amount. Dr. Griffiths' experiments as to its beneficial addition as sulphate should be carefully considered.

**Lime.**—It is needless to say this estimation is of supreme importance; if deficient—that is, under 1 per cent—and the soil fails to effervesce on addition of HCl, basic manure, that is to say, superphosphates with some insoluble or basic slag, or a dressing of lime, should be advised. Many grass lands, ever so much manured, are deficient in lime, and respond well to proper treatment.

**Potash.**—Artificial supplies may be applied when the figure is under 0.25 per cent. Of course potash is more locked up than phosphoric acid, and there is rightly a tendency to increase the use of potassic fertilisers. The cheapness of kainit, which also supplies magnesia, and so facilitates this, is a move in the right direction.

**Silicates** (insoluble in HCl).—This figure is of course of interest if excessive, over 90 per cent; it is an indication as to the need of artificial fertilisers.

The following is an example of this "partial" analysis of soil. It was from the garden of an eminent scientific friend in Kent. It may be here stated that the treatment of gardens with artificial manures is generally in a very unsatisfactory state, and it is unfortunate that those who make horticulture a calling do not give more attention to the wants of the soil instead of either totally neglecting the matter, or applying manures in an absurdly haphazard manner.

#### Partial Analysis of Garden Soil.

|                                     | Per cent. |
|-------------------------------------|-----------|
| Water .. .. .                       | 15.95     |
| Dried at 212° F.—                   |           |
| Organic matter and loss on ignition | 2.73      |
| Phosphoric acid .. .. .             | 0.08      |
| Iron and alumina .. .. .            | 1.94      |
| Lime .. .. .                        | 0.44      |
| Potash .. .. .                      | 0.05      |
| Matters not determined .. .. .      | 1.13      |
| Silica (insoluble in HCl) .. .. .   | 93.63     |
|                                     | 100.00    |

Nitrogen, 0.12 per cent

The need of artificial manure is at once apparent here, and the treatment, bearing in mind the object in view, the growth of flowers, is at once indicated. A Peruvian guano, with about 40 per cent of phosphate, 4–5 per cent ammonia, and 3–4 per cent of potash, should have excellent results.

I am quite aware there is not much novelty in the above observations, but the whole matter is so important that a few suggestions may be useful. This is a case where "a little learning" or "a little knowledge" is certainly better than "a great deal of ignorance," and if these simple and inexpensive estimations were more frequently done we should have better agricultural results, and hear less complaints of foreign competition from farmers who have not recognised that some scientific knowledge is essential to success, and by no means replaced by practical skill in agriculture.

Lawes' Works, Barking.

#### CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS.\*

ATTEMPTS TO PREPARE PRASEODYMIUM AND NEODYMIUM ALUMS—SOME NEW DOUBLE SULPHATES.

By CHARLES BASKERVILLE and HAZEL HOLLAND.

**Synopsis.**—Pure praseodymium and neodymium sulphates, i.e., free from lanthanum, were prepared and attempts made to form alums by mixing with varying proportions of the alkaline sulphates. The four methods tried, viz., concentration of solutions on the water-bath, in a vacuum desiccator, passing dry cold air over the solution kept at zero, and electrolysis (method of Piccini), failed to give an alum.

The following new double sulphates were prepared:— $\text{Pr}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ ,  $\text{Pr}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 4\text{H}_2\text{O}$ , and  $\text{Nd}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 3\text{H}_2\text{O}$ , which may serve as types.

The methods of separation of lanthanum and the didymiums were not altogether satisfactory. It was thought, as these bodies are frequently classed together, that perhaps one might form an alum and serve as a method for separating them.

The alums decrease in stability with an increase in the atomic weight of the trivalent metal, while the heavier the univalent metal, the greater the stability. For instance, sodium enters into alums with the lightest of the trivalent metals, as aluminum, vanadium, and chromium. On account of these characteristics and the statement made by Locke (*Am. Chem. Journ.*, xxvii., 281), that "if an alum cannot be formed with caesium, it cannot be formed at all," in most of our experiments caesium was used as the univalent element.

The praseodymium sulphate was prepared from oxide obtained according to the method of Baskerville and Turrentine (*Journ. Am. Chem. Soc.*, xxvi., 46; *CHEMICAL NEWS*, lxxxix., 147). The neodymium sulphate was prepared from the purest neodymium oxide obtained by the

\* Presented in abstract at the Cleveland Meeting of the American Chemical Society. From the *Journal of the American Chemical Society*, 1904, xxvi., No. 1.

method of Baskerville and Stevenson (*Journ. Am. Chem. Soc.*, xxvi., 54; *CHEMICAL NEWS*, lxxxix., 154).

No lanthanum was present in either of these preparations, so we hoped to avoid the difficulties encountered in the former paper. In fact, it has been stated that lanthanum might be removed in part from praseodymium and neodymium by heating the sulphates, which will bring about the separation of the lanthanum almost entirely free from the other two bodies. Muthmann and Rölzig made a careful comparative study of the solubility of these sulphates (*Ber.*, xxxi., 1720). While both praseodymium and neodymium sulphates are precipitated from concentrated solutions by elevation of temperature, they do not come out so readily as the lanthanum. We did not push the method to such an extent to determine if it would serve as a successful method for the separation finally, but we did learn that it would be very slow.

The praseodymium sulphate was prepared by a solution of the oxide in concentrated sulphuric acid (1:1) in platinum, the excess of acid being evaporated off by heating upon a sand-bath. The pure green crystalline praseodymium sulphate was dissolved in cold water. This standard solution contained for each cubic centimetre 0.05 gm. of the sulphate. The standard solution of caesium sulphate contained 0.039 gm. of sulphate per cubic centimetre. Equivalent amounts of these solutions were mixed and placed in a glass evaporating dish in a vacuum desiccator; within twelve hours pretty green crystals separated out. The liquor was poured off, the crystals dried with alcohol and ether, finally, by pressing between folds of bibulous paper.

The crystals were analysed according to the methods usually employed in the analysis of potash alums, namely, weighed portions were dissolved in cold water, acidified with concentrated hydrochloric acid, the praseodymium hydroxide being precipitated with ammonium hydroxide; the excess was removed by boiling; filtered, washed, and the filtrate evaporated to dryness. Caesium was determined as normal sulphate. A small amount of pure ammonium carbonate was always added after the ammonium salts had been driven off to convert the caesium-hydrogen sulphate into the normal sulphate. The praseodymium hydroxide was ignited in a platinum crucible and heated in the air with a blast-lamp to a constant weight. From a number of determinations and the amount of oxygen yielded by this greenish black residue when it was heated in hydrogen, its formula was taken as  $\text{Pr}_2\text{O}_7$ .

The first crystals obtained by the method outlined above gave, on analysis, the following:—

|                                         | Per cent. |
|-----------------------------------------|-----------|
| Praseodymium sulphate .. .. .           | 61.07     |
| Caesium sulphate .. .. .                | 35.57     |
| Water of crystallisation, by difference | 3.36      |
|                                         | 100.00    |

The body would therefore have the formula  $\text{Pr}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ —a new double salt, corresponding to the type  $\text{Pr}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ , similar to Cleve's double lanthanum sulphate and Benedick's  $\text{Gd}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$  (*Zeit. Anorg. Chem.*, xxii., 393), but it is not an alum.

As the relationship of the two sulphates was correct, an attempt was made to increase the content of water of crystallisation by mixing the sulphates at a reduced temperature; zero in the second experiment. When the mixture was kept at that temperature for sixteen hours with a rapid stream of dry air passing over it to facilitate evaporation, very soon exquisite green crystals separated out, which gave on analysis:—

|                               |       |
|-------------------------------|-------|
| Praseodymium sulphate .. .. . | 58.74 |
| Caesium sulphate .. .. .      | 37.64 |

or virtually the same compound.

The lowering of the temperature failing to raise the percentage of water, the third experiment was carried out at a temperature of 60° C. The beaker was maintained at this temperature by immersion in a carefully regulated

water-bath. The crystals separated out very quickly, and, like the others, possessed the characteristic green colour. The analysis gave—

|                               |       |
|-------------------------------|-------|
| Praseodymium sulphate .. .. . | 70.08 |
| Caesium sulphate .. .. .      | 26.99 |

While this body also contained 2 molecules of water of crystallisation, the percentage of praseodymium sulphate obtained showed the presence of either a new sulphate or that the crystals analysed constituted a mixture of those previously described and a sulphate probably resembling the nonhydrated lanthanum sulphate.

One experiment was made looking toward the preparation of the praseodymium potassium alum. Theoretical proportions of the sulphates were mixed in a glass evaporating dish and kept in a vacuum desiccator exhausted over night. Green crystals separated out. They were dried and analysed and the compound was found to be a double sulphate containing 4 molecules of water of crystallisation.

Failing to prepare the alum by simply mixing and evaporating the sulphates at different temperatures, Piccini's method of electrolysis was applied to praseodymium and caesium sulphates (*Zeit. Anorg. Chem.*, 1899, xx., 12). A description of the method is given in the previous paper. The experiment was interrupted at the end of forty-eight hours, a few crystals having separated out at the cathode. The solution was decanted and evaporated in a vacuum desiccator. The reduced pressure was continued for several days until sufficient of the crystals were obtained for analysis:—

|                                         |       |
|-----------------------------------------|-------|
| Praseodymium sulphate .. .. .           | 58.14 |
| Caesium sulphate .. .. .                | 35.67 |
| Water of crystallisation, by difference | 6.19  |

The compounds, therefore, were double sulphates with 4 molecules of water of crystallisation.

The crystals that separated out at the cathode were analysed with the following result:—

|                               |       |
|-------------------------------|-------|
| Praseodymium sulphate .. .. . | 56.73 |
| Caesium sulphate .. .. .      | 33.63 |

This is virtually the same double sulphate.

#### Attempt to prepare Neodymium Alum.

Pure neodymium oxide obtained by the method of Baskerville and Stevenson (*loc. cit.*) was converted into the sulphate in the same manner as described above for praseodymium. Standard solutions of this and caesium sulphate were prepared. Calculated quantities were evaporated *in vacuo*. Beautiful lavender crystals formed, which were analysed according to the same methods as given for praseodymium.

|                                         |       |
|-----------------------------------------|-------|
| Neodymium sulphate .. .. .              | 62.11 |
| Caesium sulphate .. .. .                | 32.34 |
| Water of crystallisation, by difference | 5.55  |

The compound was therefore a double caesium neodymium sulphate containing 3 molecules of water of crystallisation,  $\text{Nd}_2(\text{SO}_4)_3 \cdot \text{Cs}_2\text{SO}_4 \cdot 3\text{H}_2\text{O}$ .

Attempts were also made to prepare the potassium neodymium alum by electrolysis. The same method employed in preparing the praseodymium body was used. The crystals which separated out at the cathode gave, on analysis—

|                                         |       |
|-----------------------------------------|-------|
| Neodymium sulphate .. .. .              | 60.92 |
| Caesium sulphate .. .. .                | 33.08 |
| Water of crystallisation, by difference | 6.00  |

This body was also a double sulphate containing 3 molecules of water of crystallisation.

Aluminum salts possess weak basicity, while the praseodymium and neodymium are, comparatively, basic (*Am. Chem. Journ.*, xxvi., 166). This may account in part for our failure to prepare these alums. It is particularly interesting to note in considering these two so-called elements that the one gives a series of double salts with 2 and 4 molecules of water of crystallisation, while the other gives one with 3.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.

By CHARLES BASKERVILLE, Ph.D., F.C.S., Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 171).

## LIST OF PROPOSED CHEMICAL ELEMENTS (continued).

| Date.        | Name.                                                                                    | Source.                                                                                    | Authority.                                  | Reference.                                                                                      | Remarks.                                                                                                                                                                                                                                                                                                                                                                                                                                                  |
|--------------|------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------|---------------------------------------------|-------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1887         | Era.<br>Erß.<br>Tma.<br>Tmg.<br>Sma.<br>Smß.<br>Xa to X <sub>μ</sub> (7).<br>Div to Dia. | Erbia.<br>Erbia.<br>Thulia.<br>Thulia.<br>Samarita.<br>Samarita.<br>Soret's X.<br>Didymia. | Krüss and Nilson.<br>Kiesewetter and Krüss. | Ber., 20, 2134; 21, 2310.                                                                       | Lettered according to source; the Greek characters serve to show each one has one absorption line or band. This is an argument as to the complexity of those elements showing absorption spectra. A most careful study of these and other data offered by Schützenberger, Boudard, Crookes, Brauner, Muthmann, Dennis, Baskerville, and others enforces the "one band one element" idea of Crookes. Distinctly, however, so far the case is "not proven." |
| 1887         | Three unknown new earths.                                                                | Didymium from cerite.                                                                      | Demarcay.                                   | Comptes Rendus, 102, 1552.<br>Comptes Rendus, 104, 580.<br>Comptes Rendus, 102, 1552.           | Absorption band $\lambda$ 475 in didymium spectrum (also Crookes, Comptes Rendus, 102, 1551).<br>Band $\lambda$ 462 in old didymium spectrum; same giving rise to 475, belong to samarium series according to Crookes (see). Absorption band $\lambda$ 434 in old didymium spectrum.                                                                                                                                                                      |
| 1889         | Russium.                                                                                 | Thorium from monazite.                                                                     | Chroustschoff.                              | Berg. u. Hüt. Zeit., 4, 329;<br>Chem. News, 59, 234.                                            | Meagre information as to procedure to be had. Prepared in same manner as Barriere obtained lucium. From many alumina preparations.                                                                                                                                                                                                                                                                                                                        |
| 1889         | Austriacum.                                                                              | Tellurium.                                                                                 | Brauner.                                    | Trans. Chem. Soc. (Lond.),<br>407, 411; Monats., 10, 448.                                       | Working on atomic weight of tellurium, Brauner thought he secured homologue atomic weight 214. Later modified claims, stating homologue of argon (Journ. Chem. Soc., 67, 599). See Kötner (Annalen, 1890, 319, 1). Staudenmaier (Zeit. Anorg. Chem., 1895, 10, 180) and Norris, Fay, and Edgerly (Am. Chem. Journ., 1900, 23, 105) concluded tellurium elementary.                                                                                        |
| 1889         | Eka-Tellurium.                                                                           | Tellurium, antimony, and copper.                                                           | Grünwald.                                   | Imperial Academy of Sciences,<br>Vienna, 98, 2; Chemical<br>News, 61, 39.                       | Characteristic unknown element X in 11th series of Mendeleeff's table, related to tellurium on one hand and bismuth on other. Probable atomic weight 212 and identical with Brauner's austriacum. Wave-lengths of 16 rays observed in ultra-violet between 2729—2159. See Ames' remarks re Grünwald's helium and coronium conclusions.                                                                                                                    |
| 1890<br>1892 | Damarium.<br>Gnomium.                                                                    | Cobalt and nickel.                                                                         | Much.<br>G. Krüss and F. W. Schmid.         | Chem. Zeit., 1890.<br>Berichte, 1889, 22, 11; 22,<br>2026; Zeit. Anorg. Chem.,<br>1902, 2, 235. | Disproved by Winkler (Zeit. Anorg. Chem., 1893, 4, 10).<br>Good fun.                                                                                                                                                                                                                                                                                                                                                                                      |
| 1892         | Marrum.                                                                                  | Egyptian Johnsonite or maserite.                                                           |                                             | Chem. Zeitung; Chemical<br>News, 65, 259.                                                       | Said to have atomic weight 228, apparently no need to confirm this (Crookes).                                                                                                                                                                                                                                                                                                                                                                             |
| 1892         | Za.                                                                                      | Samaraskite and gadolinite.                                                                | De Boisbaudran.                             | Comptes Rendus, 119, 575.                                                                       | Follows samarium spark spectrum; see Europium, (Comptes Rendus, 1469).                                                                                                                                                                                                                                                                                                                                                                                    |
| 1892         | Zç.                                                                                      | Terbia.                                                                                    | Hofmann, G. Krüss.                          | Zeit. Anorg. Chem., 4, 27.                                                                      | Follows samarium reversion bands. Demarcay's europium.                                                                                                                                                                                                                                                                                                                                                                                                    |
| 1893<br>1894 | Probable earth.<br>A new element (?).                                                    | French bauxite.                                                                            | K. I. Bayer.                                | Chem. Zeit., 18, 671.                                                                           | Based on some qualitative tests.                                                                                                                                                                                                                                                                                                                                                                                                                          |

|      |                         |                            |                               |                                                                |                                                                                                                                                                                                                                                                                                                                                                                                                                               |
|------|-------------------------|----------------------------|-------------------------------|----------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1895 | Zs.                     | Terbia.                    | De Boisbaudran.               | Comptes Rendus, 121, 709 ;<br>Chemical News, 72, 292.          | Paper in sealed packet deposited with the French Academy. Beginning of Crookes-de Boisbaudran spirited controversy. Absorption band $\lambda$ 487.7 does not belong to terbium or dysprosium.                                                                                                                                                                                                                                                 |
| 1895 | Arcoon.                 | Atmosphere.                | Rayleigh and Ramsay.          | Roy. Soc. London, January 31, 1895.                            | Holds its own although numerous efforts have been made to show it to be allotropic nitrogen. Distinctly inert element. A hydrate has been suggested. Also separated by Cavendish (Phil. Trans., 1785, 75, 372; 1788, 78, 271).                                                                                                                                                                                                                |
| 1895 | Per-Helium.             | Helium.                    | Runge and Paschen.            | Math. nat. Mitt., Berlin, 323.                                 | Found lines in spectrum could be divided into two sets, and each set consisted of a primary and two secondary series of lines. Later found change of pressure in tube will cause this change—as with oxygen, for example.                                                                                                                                                                                                                     |
| 1896 | Lucium.                 | Yttria from monazite.      | Barriere.                     | Chemical News, 74, 159, 212.                                   | Shown to be a mixture of yttrium, didymium, erbium, and terbium by Crookes (Chemical News, 74, 259) and Shapleigh (Chemical News, 75, 41).                                                                                                                                                                                                                                                                                                    |
| 1896 | "Σ" or "Σ-Z."           | Gadolinitium and samarium. | Demarçay.                     | Comptes Rendus, 122, 728 ;<br>132, 1484.                       | Extreme fractionation of double magnesium nitrate; atomic weight 151.5. Most characteristic ray, Crookes's anomalous $S^2$ (fluorescent) and $Z\epsilon$ of de Boisbaudran (reversion) spectrum (Comptes Rendus, 1901, 132, 148). See Europium.                                                                                                                                                                                               |
| 1895 | Unnamed.                | Monazite.                  | Schützenberger and Boudouard. | Comptes Rendus, 121, 273 ;<br>122, 697 ; 123, 782 ; 126, 1648. | Fractional fusion of nitrates and crystallisation of sulphates obtained. Atomic weight, 124 (?). Urbain states this is mixed with other rare earths (Bull. Soc. Chim., [3], 19, 38). See Lucium; also Norden-skjöld's "oxide of gadolinium" (Comptes Rendus, 1903, 795).                                                                                                                                                                      |
| 1896 | Kosmium.<br>Neokosmium. |                            | Kosmann.                      | Zeit. f. Electrochemie, 1896-7.                                | Winkler remarks ("Discovery of New Elements, &c.") German Chemical Society, January 11, 1897:—"If it were not for the expense of the patent, it might have been thought a plesantry." But see Barriere's Lucium.                                                                                                                                                                                                                              |
| 1896 | Unnamed.                | Monazite.                  | Drossbach.                    | Berichte, 29, 15 ; 30, 2452.                                   | Having worked up a large amount of the by-products obtained in extracting thorium from monazite, obtained strongly basic earth, sulphates soluble in double alkaline sulphates. Atomic weight, 100 (?). Boudouard and Schützenberger obtained one with 102. Urbain and Budischovsky (Comptes Rendus, 124, 618) fractionated these by acetylacetone compounds in organic solvents into extremes giving values 95 and 112. Mixture. See Lucium. |
| 1897 | Metacerium.             | Cerium.                    | Brauner.                      | Proc. Chem. Soc., No. 191, 69-70 ; Chemical News, 71.          | By fractioning what was thought to be pure ceria, differences in specific gravity oxides, atomic weight, but no spark spectral differences. Perhaps has atomic weight 110, according to Brauner.                                                                                                                                                                                                                                              |
| 1897 | Glaukodidymium.         | Didymium.                  | Chroustschoff.                | Journ. Russ. Phys. Chem. Soc., 29, 206.                        | Distinguished from praseodidymium and neodidymium by blue colour of salts. (See these elements ; no doubt as to their complexity).                                                                                                                                                                                                                                                                                                            |
| 1897 | A possible new element. | Cast-iron.                 | Boucher.                      | Chemical News, 76, 99.                                         | See also Rubdock (Chemical News, 76, 118). Thought to be molybdenum by C. H. Jones (Chemical News, 76, 171). Denied by Boucher.                                                                                                                                                                                                                                                                                                               |

(To be continued).

## PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

ANNUAL GENERAL MEETING, *March 23rd, 1904.*

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

DR. G. BARGER and Dr. S. B. SCHRYVER were appointed scrutators, and the ballot was opened for the election of Officers and Council for the ensuing year. The PRESIDENT then presented the following Report on the state of the Society during the past twelve months:—

*Report of the Council.*

The Council are in the fortunate position of being able to report that the Society continues to increase and flourish. At the date of the Annual General Meeting last year, the number of Fellows was 2631 (last year this number was incorrectly stated to be 2471); since that date, 170 Fellows have been elected and three reinstated, making a gross total of 2804. Of these, 23 have been removed for non-payment of subscriptions, and the elections of 2 have become void, 28 have resigned, and 17 have died, leaving a net total of 2734 Fellows on our list.

The names of the Fellows who have died are:—H. G. Adshead, J. O. Alexander, A. E. Barrows, Baron de Bush, Samuel Clift, W. H. Corfield, T. H. Dodd, T. W. Fletcher, W. Francis, H. B. Fulton, A. G. Hendry, T. Isherwood, C. James, T. A. Lawson, J. Mactear, J. Reddrop, G. H. Robertson.

As in future Reports the accounts and other statistics of the Society will be given for the calendar year, the following data for the year 1903 may be of interest for purposes of comparison. The number of Fellows on January 1st, 1903, was 2607, and during the year 173 Fellows have been elected and 5 reinstated by the Council, making a gross total of 2785. Of these, the Society has lost 16 by death, 46 from resignations, and 23 by removal for non-payment of subscriptions, giving the number of Fellows in the Society on December 31st, 1903, as 2700.

The Society has to lament the death of a distinguished Foreign Fellow, Professor Wladimir Markownikoff, on February 11th, 1904.

The small number of Fellows elected in the early days of the Society has been further reduced by the death of Dr. William Francis.

The scientific work of the Society during the past session gives evidence of continued activity. Since the last Annual General Meeting, 163 scientific communications have been made to the Society, 107 of which have already been published in the *Transactions*, and abstracts of all have appeared in the *Proceedings*.

During 1903, 181 scientific communications were made to the Society, 110 of which were published in the *Transactions* for that year, abstracts of all appearing in the *Proceedings* for 1903.

The *Journal* for 1903 contains also 3882 abstracts, which may be classified as follows:—

|                                                           | PART I. | Pages. | No. of Abstracts. |
|-----------------------------------------------------------|---------|--------|-------------------|
| Organic Chemistry .. .. .                                 | 872     | 1650   |                   |
| General and Physical Chemistry .. .. .                    |         | 471    |                   |
| Inorganic Chemistry .. .. .                               |         | 443    |                   |
| Mineralogical Chemistry .. .. .                           |         | 119    |                   |
| Physiological Chemistry .. .. .                           |         | 391    |                   |
| Chemistry of Vegetable Physiology and Agriculture .. .. . |         | 242    |                   |
| Analytical Chemistry .. .. .                              |         | 566    |                   |
|                                                           | 768     | 2232   |                   |
| Total in Parts I. and II. ..                              | 1640    | 3882   |                   |

The volume of *Transactions* for 1903 contains 142 memoirs occupying 1490 pages, whilst that for the preceding year contains 160 memoirs occupying 1604 pages.

In April last, Professor Emil Fischer was invited to give the Faraday Lecture in the autumn, and provisional arrangements were made for its delivery early in the present session. Much to the regret of the Council, continued ill-health has prevented Professor Fischer from fulfilling his intention, and for the present the hope of numbering him among the Faraday Lecturers has had to be abandoned. The Council are glad to be able to announce that Professor Ostwald will deliver the Faraday Lecture on Tuesday, April 19th, in the Lecture Theatre of the Royal Institution, kindly lent by the Managers for the occasion.

The Centenary of the Enunciation of the Atomic Theory by Dalton was celebrated by the Literary and Philosophical Society of Manchester in May last, and in company with Sir Henry Roscoe, Professor Thorpe, and Professor Frankland, Vice-Presidents, Dr. Scott, Secretary, and Sir William Ramsay, Foreign Secretary, the President attended the meeting, and presented an address in the name of the Chemical Society.

The triennial award of the Longstaff Medal to the Fellow of the Society who, in the opinion of the Council, has done the most to promote chemical science by research in the interval since the last presentation, was made, on the recommendation of the Research Fund Committee, to Professor W. J. Pope, F.R.S., for his researches on the stereochemistry of compounds of elements other than carbon.

The Fifth International Congress of Applied Chemistry was held in Berlin in June last, and the President attended as the representative of the Society. An invitation to the Congress to hold its next meeting in London, given by the President of the Society of Chemical Industry, was supported by the President, but on a vote being taken Rome was chosen as the place of the meeting in 1905.

The Council welcomed the opportunity of sending a letter of congratulation to their distinguished Foreign Fellow, Professor Mendeleeff, on the occasion of his seventieth birthday, which occurred on February 9th last.

The increase in the use of the Library mentioned in last year's report has been maintained, as 975 books were borrowed from the Library, as against 925 during the previous year. The additions to the Library comprise 115 books, of which 49 were presented, 268 volumes of periodicals, and 48 pamphlets; as against 84 books, 338 volumes of periodicals, and 23 pamphlets last year. The corresponding numbers for the year ending December 31st, 1903, are 991 books borrowed, the additions to the Library being 126 books, of which 51 were presented, 271 volumes of periodicals, and 43 pamphlets.

The changes proposed in last year's report with regard to the Library have now been carried out, and cases have been erected in one of the rooms in the basement for the storage of the less used books. These cases will accommodate about 3500 volumes, and so provide room for the growth of the Library for about seven years. An equal number of cases can be added at any future period without cost beyond that of the cases themselves.

Mr. F. W. Clifford was appointed Librarian on July 1st.

The Council regret to have to record the death, on May 10th, of Mr. Josiah Hall, who for twenty-five years had been Collector to the Society, and who retired in 1895 with a pension of £130 a year. It was decided to grant to Mrs. Hall, his widow, an annuity of £30.

The Society has been the fortunate recipient of two handsome busts in bronze of distinguished chemists, that of Liebig (a copy of the one in Munich) having been presented by Dr. Messel, and that of Dalton, which has been modelled from all available sources by Miss Levick, by Professor Thorpe. Two interesting photographs of portraits have also been received, one of Roger Bacon, presented by Mr. Oscar Guttman, and one of Dr. Wm. Prout, F.R.S., from his son, the Rev. T. J. Prout.

At an Extraordinary General Meeting of the Society held

On July 2nd, the proposal of the Council to alter By-law I. so that the annual publications of the Society should not be sent to Fellows who are in arrear with their subscriptions, was unanimously approved. To give effect to this change in the By-law, the *Journal* is now issued to Fellows on the last day instead of the first day of the month. An extra number of the *Transactions* was issued on December 31st to provide for the publication of papers which would have appeared on January 1st, 1904, under the old arrangement, and now that the change has been made the net result is that Fellows receive the monthly parts of the *Journal* a day earlier than heretofore.

The first part (Authors' Index) of the Collective Index for the decade 1893-1902, promised provisionally for 1904, was issued in January to those Fellows who had made application for it in accordance with the printed notices circulated with the monthly parts of the *Journal* since last July, and it is hoped that the subject index for the same period may be ready before the end of next year. The rapid publication of the Authors' Index is due to the untiring energy and devotion of the Indexer, Mrs. Dougal, and has in no way interfered with the issue of the Annual Index at the end of February, which has been customary since she undertook the work involved in its preparation.

The Second Report of the Joint International Committee on Atomic Weights, which now includes Professor Moissan as the representative of France, with its revised table of atomic weights, has been issued to Fellows both in the *Proceedings* and in the *Journal*.

The Council resolved that the rooms of the Society should be closed not later than 10.30 p.m. on those evenings on which meetings are held, and, as very few Fellows were found to use the Library on Saturday afternoons, it was further resolved that it should be closed at 2 p.m. on Saturdays from the beginning of the present year.

Grants amounting in all to £232 have been made during the year from the Research Fund, of which amount £15 has been returned.

The Council desires to record its deep sense of obligation to Dr. Horace T. Brown for the ability and patience with which he has discharged the duties of Treasurer during the past year. Finding on taking office that a complete re-organisation of the method of collecting the contributions of Fellows and of keeping the Accounts of the Society was immediately necessary, he did not shrink from the immense labour involved in establishing a new system and getting it into operation. The nature of the changes which have been made is explained below, and it is obvious that great sacrifices of time and of personal effort were involved in the process. It is therefore with the utmost regret that the Council has received from Dr. Brown an intimation that his engagements no longer permit him to retain office.

Hitherto, it has been customary for the Treasurer to close the Books of the Society a few days before the Annual General Meeting, and to make up the Accounts to a date as near to that of the Annual Meeting as possible. This arrangement has given rise to considerable inconvenience, owing, in the first place, to the short time allowed to complete the financial statements, and, secondly, to the impossibility of conforming strictly to that portion of By-law VIII. which requires the Auditors to report to the Council at least one week before the Annual General Meeting. In future, therefore, the Treasurer will present his Accounts at the Annual Meeting made up to December 31st, instead of to some indeterminate day near the end of March, and these Accounts will be drawn up in a somewhat different form from that previously adopted. A statement of the financial condition of the Society made up for the year ending December 31st last in this modified form accompanies this Report, including a Revenue and Expenditure Account and Balance Sheet, but the Treasurer has considered it advisable on this occasion to include for purposes of comparison another statement made out on exactly the old lines up to March 12th, and it is to this

latter statement, which is comparable with the one of last year, that the following remarks apply:—

The total income of the Society, from March 22nd, 1903, to March 12, 1904, was £7276 16s. 5d., whilst the expenditure for the same period was £6060 17s. 4d., showing an excess of income over expenditure of £1215 19s. 1d., against a surplus last year of £817 7s. 1d. The comparison, however, is not a fair one, unless we adjust the figures in the respective years for the arrears of contributions in each case.

Owing to the alteration in July last of By-law I., by which Fellows who are in arrear with their subscriptions do not receive the publications of the Society, the annual contributions, which become due in January, have been paid up more punctually this year than usual. On March 22nd, 1903, the arrears amounted to £2316, whilst on March 12th this year they were £2008, a difference in favour of this year of £308. When this adjustment is made, the surplus of this year becomes £907 19s. 1d. against £817 7s. 1d. last year, a result which may be regarded as highly satisfactory when we take into consideration certain large items of extraordinary expenditure which have been incurred this year, and which will be alluded to later.

The total amount received this year for Admission Fees, Life Compositions, and Subscriptions is £5809 against £4776 last year, a difference in favour of this year of £1033. This, however, includes £250 received on Account of Arrears of Subscriptions from the late Assistant Secretary, and, as already stated, the subscriptions this year are paid up more closely by £308. With these adjustments, the increased income this year from contributions of Fellows is £475, about half of which is due to the unusually large number of Life Composition Fees.

The sale of the Society's publications, and the proceeds of the advertisements in the *Journal* have brought in £40 19s. 4d. more than last year.

With regard to investments, the Treasurer has to report that since the last General Meeting he has, with the authority of the Council, purchased on behalf of the Society £1500 of Transvaal 3 per cent Guaranteed Stock. The investments on the General Account now stand at £18,909 os. 5d., whilst those of the Research Fund Account are valued at £6659 5s. 5d.

The total expenditure this year shows the large increase of £665 19s. 4d., which is due to certain large items of an exceptional nature.

The balance of the cost of the first volume of the Decennial Index, 1893-1902, amounting to £386 9s. 7d., is chargeable against this year's revenue, being an increase of £254 5s. 3d. The completion of the Library Catalogue was announced in the Report of last year, but the main cost of £156 16s. 0d. has been borne by the present accounts. The additional accommodation for books referred to in an earlier part of this Report has been provided at a cost of £128 2s. 4d. Two other large items of expenditure of an exceptional nature amounting to £228 1s. 6d. are included under Accountants' and Legal charges, and are mainly attributable to a special investigation of the Society's Accounts to 1895, to which reference has already been made.

It is satisfactory to notice that the expenses incidental to the publication of the *Journal* show a decrease this year of about £100.

The system of keeping the Treasurer's accounts has undergone revision during the past year, the old system having been found insufficient for the present needs of the Society.

At the request of the Council, Messrs. W. B. Keen and Co., Chartered Accountants, have opened up a new set of books, and have also undertaken to give the Treasurer a quarterly audit, and to assist in the preparation of the accounts for the Annual Statement.

This arrangement, which guarantees skilled professional assistance and a thorough checking of the accounts at frequent intervals, has now been in operation for some time, and will not entail any additional expense to the

Society, as the appointment of a Treasurer's Assistant is thereby rendered unnecessary.

Arrangements have been made with Messrs. Coutts and Co., the Society's Bankers, by which in future they will receive the contributions of Fellows direct.

Prof. H. B. DIXON moved the adoption of the report, which was seconded by Mr. E. W. VOELCKER, and carried unanimously.

Dr. SCOTT proposed, and Dr. PHILIP seconded a vote of thanks to the Auditors, which was acknowledged by Dr. MOODY.

The PRESIDENT then delivered his Address, which was devoted chiefly to a review of the scientific history of the Society since its foundation in 1841. The early volumes of the *Memoirs of the Society* contain papers by many eminent foreign chemists, such as that on "The Atomic Weight of Carbon," by Professors Redtenbacher and Liebig, and the memoir on "The Radical of the Cacodyl Series," by Professor Bunsen. These and others, such as Frankland's paper on "The Isolation of the Organic Radicals," indicate the general nature of the problems which occupied chemists in the early days of the Society. The *Quarterly Journal*, which, in 1849, succeeded the three volumes of *Memoirs*, contain many contributions by Hofmann, Williamson, Frankland, Kolbe, and Odling, but as it was the custom in those days for authors to send all their serious work to the Royal Society, the *Journal of the Chemical Society* does not provide a continuous picture of the results of chemical investigation in this country. In 1862, the *Quarterly Journal* was replaced by a monthly issue, and in 1863 a new series was commenced.

At that period, language and formulæ were alike governed by the then predominant theory of types. Modern views of constitution based on clearer ideas of valency were not definitely exhibited in the *Journal* till 1865, when Dr. Crum Brown's paper appeared on "The Theory of Isomeric Compounds." The change could, however, be accomplished only after agreement had been arrived at in reference to atomic weights. Stereochemical ideas, inaugurated in 1875 by Le Bel and van't Hoff, have reached their logical conclusion in the work of Pope.

Other subjects traced through the successive volumes of the *Journal* are the spectroscopic, the periodic scheme of classification, molecular weights, and the application of Avogadro's law. The paucity of systematic research in the United Kingdom during so many years was referred to, and some of the causes which led to the publication of so few papers by the Society, and the rapid development of activity during the last twenty years.

The Address concluded with a suggestion for the publication by the Society of annual reports on the progress of chemistry in its several more important divisions.

Prof. EMERSON REYNOLDS proposed a vote of thanks to the President, coupled with a request that he would allow his Address to be printed in the *Transactions*.

Mr. DAVID HOWARD seconded the motion, which was carried by acclamation, and acknowledged by the PRESIDENT.

Prof. DIVERS proposed a vote of thanks to the Treasurer, Secretaries, and Council. This was seconded by Mr. SPILLER and unanimously adopted. Dr. SCOTT responded.

The Scrutators presented their report to the President, who declared the following to have been duly elected as Officers and Council for the ensuing year:—

*President*—W. A. Tilden, D.Sc., F.R.S.

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### Errata.

P. 141, col. 2, the formula at foot should read " $\text{CO}_2\text{H}\cdot\text{CH}<$ ," &c. P. 142, col. 1, line 21, read "*cis-a-hydroxyhexahydroterephthalic acid*."

### The Faraday Lecture.

The Faraday Lecture will be delivered by Prof. W. Ostwald on Tuesday, April 19th, 1904, at 8.30 p.m.

Prof. Ostwald will lecture in English, the subject of his discourse being "Elements and Compounds."

The lecture will be delivered, by the kind permission of the Managers, in the Theatre of the Royal Institution, 21, Albemarle Street, W.

## NOTICES OF BOOKS

*Mineral Tables for the Determination of Minerals by their Physical Properties.* By ARTHUR S. EAKLE, Ph.D. First Edition, First Thousand. London: Chapman and Hall, Limited. New York: John Wiley and Sons. 1904. Pp. 73.

THESE tables will be found very useful for reference in the determination of minerals by means of their physical properties; they include the common minerals and a few others of local prominence which are generally looked upon as rare. The minerals are arranged primarily according to streak and "color" (the book being printed in the United States), and under each "color" the arrangement is according to hardness. The other properties tabulated are composition, lustre, system of crystallisation, cleavage or fracture, specific gravity, and common structure, with a final column for remarks and observations. There are a few preliminary pages describing all the properties in detail, and giving an analytical key; there is also a full index.

*X-Rays Simply Explained: a Handbook on the Theory and Practice of Radiography.* By R. P. HOWGRAVE-GRAHAM, Assoc.I.E.E. Fully Illustrated, with Six Full-page Plates. London: Percival Marshall and Co. Pp. 93.

WHILE intended specially for students and amateurs, this book will also be of use and interest to the practical man. As the author very wisely puts it, "the mere production of fairly good radiographs does not entitle a man to call himself practical; unless he understands some of the principles which underlie the work he is more hopelessly unpractical than he who is frankly and modestly ignorant." In these pages the author gives a thorough description of the principles involved in Röntgen, or X-ray, work, and of the use and management of the apparatus employed, including the taking of radiographs and the construction of fluorescent screens. At the end of the book there are half-a-



dosen good radiographs illustrating different methods of procedure, but there is no index.

*A Treatise on the Theory of Solution; Including the Phenomena of Electrolysis.* By W. C. D. WHETHAM, M.A., F.R.S. Cambridge: At the University Press. Pp. 488.

As the use of modern thermodynamic methods, founded on the investigations of Willard and Gibbs, is extending to almost all branches of this subject, and as such methods are still unfamiliar to many students of chemistry and physics, the author has very wisely written an introductory chapter explaining the thermodynamic principles which are applied in the body of the work. In succeeding chapters he deals with the phase rule and its applications to systems of one, two, and three components.

The general problem of solubility is first dealt with in Chapter IV., and in Chapter VII. we come to a consideration of the theories of solution; with regard to the conclusions arrived at by the author as to the nature of solution, he points out that there are two different views, each offering a reasonable explanation of the phenomena. He says:—"At first sight the idea of molecular bombardment on the walls of the membrane by solute particles which are dynamically independent of the solvent molecules seems diametrically opposed to the hypothesis of chemical combination between them; but we know too little about the nature of chemical affinity to be quite sure that it is not due to some relation in the dynamical properties of the reagents, and the two views of solution may after all be different statements of the same truth."

In Chapter VIII. the subject of electrolysis is attacked, commencing with its history, Faraday's work, and the nature of ions. The conductivity of electrolytes and the nature of galvanic cells form the subjects of Chapters IX. and X., and in Chapter XII. we come to the theory of electrolytic dissociation. The final chapters on "Diffusion in Solutions" and "Solutions of Colloids" complete a work which will rank among the foremost of those written on this subject. It is to a great extent mathematical, but the theories and hypotheses put forward by the author are dealt with in a very clear and interesting manner, so that those who are not really mathematicians can follow the subject with profit and pleasure.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxviii., No. 11, March 14, 1904.

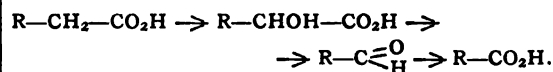
**Solubility of Silicon in Zinc and Lead.**—Henri Moissan and F. Siemens.—Silicon dissolves in zinc at a much lower temperature than in lead. The solubility starts at about 550°. At a temperature of 850°, 1.62 per cent is dissolved. An examination of the form of the curve of solubility shows that about 850° the solubility increases very rapidly, but the authors' experiments would not allow of concordant determinations above this temperature—the boiling-point of zinc being 920°, according to M. Berthelot. In the case of lead, the solubility of silicon starts at a higher temperature, 1100°, and even at 1400° only 0.15 per cent is dissolved. Finally, at the boiling-point of lead, only 0.79 per cent is dissolved. The authors also find that the silicon separates on cooling in the form of crystals possessing all the properties of this metalloid, and whose density is constant and very near the normal density of silicon.

**A New Method of Preparation of Calcium Carbide.**—Henri Moissan.—In all former experiments the author proved that carbon only reduces lime at a very high temperature, when the latter substance has just attained the liquid state. This condition necessitates the use of the electric furnace for the commercial preparation of calcium carbide. He proves, however, that metallic calcium combines with lamp-black at a dull red heat to form pure calcium carbide, crystalline and transparent. His present research is on the most convenient method of producing the calcium required in this preparation by the electrolysis of calcium chloride or of a mixture of chloride and fluoride of calcium.

**An Apparatus for Regulating the Action of Vacuum Pumps.**—J. Meunier.—The author draws and describes an apparatus for regulating the action of vacuum pumps.

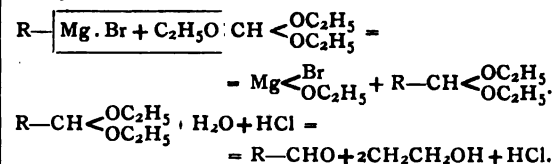
**Action of Carbonic Acid on Solutions of Sodium Nitrite.**—C. Marie and R. Marquis.—By various experiments on the action of carbon dioxide on solutions of sodium nitrite, the authors find that, in a solution of sodium nitrite containing carbonic acid, there is always a certain amount of free nitrous acid present. Even though the quantity is extremely small, too small to affect the ordinary tests for the acid, the fact remains that this mixture conforms to the general laws of the equilibrium between electrolytes (action of an acid on the solution of a neutral salt).

**Method of Preparation of Aldehydes, and the Methodic Degradation of Acids.**—E. E. Blaise.—The author finds that the preparation of aldehydes by decomposition of  $\alpha$ -oxyacids under the influence of heat is perfectly general, as he shows by various examples, and can be employed for the preparation of all aldehydes containing  $C_n-1$ , corresponding to a fatty acid containing  $C_n$ . In particular, he applies this method to the preparation of aldehydes of moderate and high atomic weight, to obtain which the corresponding primary alcohol does not constitute an accessible primary substance. Finally, the distillation of  $\alpha$ -oxyacids constitutes the best method of methodic degradation of acids by the following series of reactions:—



The advantage of this method of degradation consisting in its easy execution, in the easy isolation in the pure state of the intermediate products, and finally in the good yields obtained.

**A General Method of Synthesis of Aldehydes.**—F. Bodroux.—In a preceding research the author showed that the action of ethyl-ortho-formate on aromatic organo-magnesium compounds in ethereal solution give rise to the aldehydes—



He applies these reactions to various compounds, and finds this a convenient method of synthesising aldehydes.

**Certain Derivatives of  $\alpha$ -Campholytic Acid and  $\alpha$ -Campholenic Racemic Acid.**—G. Blanc and M. Desfontaines.—Continuing the experiments made by M. Blanc, both alone and in collaboration with M. Blaise, to prove  $\alpha$ -campholenic and  $\alpha$ -campholytic acids to be homologous, the authors now prepare the campholytic nitrile, unknown up to the present time. They then reduce the nitrile and compare the base obtained with  $\alpha$ -racemic aminocampholene. Apparently these two bases are dif-

ferent. When the two ureas and the two oxamides are mixed, after crystallisation they obtain products which melt respectively at  $114^{\circ}$  and  $130^{\circ}$ . It therefore appears that the base obtained by reduction of the  $\alpha$ -campholytic nitrile is impure  $\alpha$ -aminocampholene, the nitrile not being a homologous body.

## MISCELLANEOUS.

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 11th inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. The Right Hon. the Marquis of Salisbury, Mr. W. B. Anderson, Mr. J. Benson, Mrs. G. E. Bröun-Morison, Mrs. Douglas Cow, Mrs. J. Mackenzie Davidson, Mr. J. A. W. Dollar, Mr. Bayntun Hippisley, Mr. E. W. Linging, Mrs. Master, Mr. J. C. Prince, Mr. E. A. Short, Mr. H. L. Tidy, Mr. W. Watson-Taylor, and Mr. C. S. Whitehead were elected members. The special thanks of the members were returned to Mr. Francis Gaskell for his donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures.

**Institute of Chemistry of Great Britain and Ireland.**—The next Examinations for persons desirous of qualifying themselves to be public and technical analysts, will be held in July, 1904. The Examinations are open only to candidates who have complied with the regulations. The Intermediate Examination will be held in the Laboratories of the Institute, commencing on Tuesday, the 5th of July. Examinations in the following branches of the Final Examination for the Associateship (A.I.C.) will also be held in the Laboratories of the Institute, commencing on either Tuesday, the 5th, or Tuesday, the 12th of July:—(a) Mineral Chemistry, (b) Metallurgical Chemistry, (c) Physical Chemistry, (d) Organic Chemistry, and (e) The Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy. Applications for admission to the July Examinations should be forwarded to the Registrar not later than Tuesday, the 7th of June, 1904, on which day the List of Candidates will be closed.

## MEETINGS FOR THE WEEK.

**TUESDAY, 19th.**—Royal Institution, 5. "The Transformations of Animals," by Prof. L. C. Miall, F.R.S.  
Society of Arts, 8. "The Sentiment of Decoration," by Al red East, A.R.A.

**WEDNESDAY, 20th**—Society of Arts, 8. "Motor Cars for Popular Use," by Mervyn O'Gorman, M.Inst E.E.  
Microscopical, 8. Exhibition of Pond Life.  
Chemical, 5.37. "Vapour Density of Hydrazine Hydrate" and "Combining Volumes of Carbon Monoxide and Oxygen," by A. Scott. "Ammoniacal Double Chromates and Molybdates" and "Double Chromates of the Series  $M_2M'(CrO_4)_2 \cdot 6H_2O$ —Magnesium and Nickel Compounds," by S. H. C. Briggs. "Experiments on the Synthesis of the Terpenes—Part I, Synthesis of Inactive Terpeneol, of Dipentene, and of Terpin Hydrate," by W. H. Perkin, jun. "A Lævo-rotatory Modification of Quercitol," by F. B. Power and F. Tutin. "Constituents of the Essential Oil of Californian Laurel," by F. B. Power and F. H. Lees. "Some Derivatives of Umbellulone," by F. H. Lees.

**THURSDAY, 21st.**—Royal Institution, 5. "Dissociation," by Prof. Dewar, F.R.S., &c.

**FRIDAY, 22nd.**—Royal Institution, 5. "Sleeping Sickness in Uganda," by Col. David Bruce, R.A.M.C., F.R.S.  
Physical, 5. "Calculation of Colours for Colour Sensitometers, and the Illumination of 'Three Colour' Photographic Transparencies by Spectrum Colours," by Sir W. de W. Abney, F.R.S. "On Normal Filing as connected with Osborne Reynolds's Theory of the Universe," by Prof. J. D. Everett, F.R.S. "Note on the Diffraction Theory

of the Microscope as applied to the case when the Object is in Motion," by Dr. R. T. Glasbrook, F.R.S. Exhibition of Apparatus by Peter Heele.  
**SATURDAY, 23rd.**—Royal Institution, 3. "Cameos," by Cyril Davenport, F.S.A.

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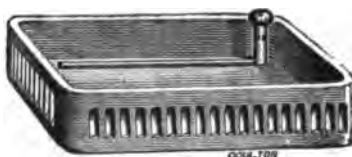
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## CONTENTS.

| ARTICLES:—                                                                                                                                     | PAGE |
|------------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Solubility of some Salts of the Lower Fatty Acids, by H. Stanley, B.Sc. (Lond).                                                            | 193  |
| The Elements—Verified and Unverified, by Charles Bankerville, Ph.D., F.C.S.                                                                    | 194  |
| Observations on some Continuous Intramolecular, and at first Reversible, Changes extending over Prolonged Periods of Time, by R. J. Friessell. | 196  |
| Note on Scale for Chemical Valuation of Soils, by Vincent Edwards, F.I.C., F.L.C.                                                              | 197  |
| A New Process of Preparing Venetian Red, by John Geddes McIntosh.                                                                              | 197  |
| Contributions to the Chemistry of the Rare Earths, by Chas. Bankerville and George F. Catlett.                                                 | 197  |
| Chemical Equilibrium between the Ferro- and Ferri-hydrocyanic Acids, by Maurice Prud'homme.                                                    | 199  |
| Examination of some Ancient Samples of Bread, by L. Lindet.                                                                                    | 200  |
| THE FARADAY SOCIETY                                                                                                                            | 200  |
| NOTICES OF BOOKS                                                                                                                               | 201  |
| CORRESPONDENCE                                                                                                                                 | 202  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                          | 202  |

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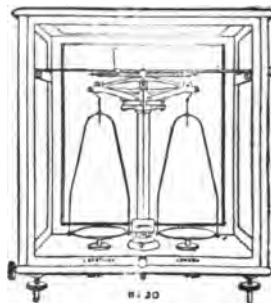
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# THE CHEMICAL NEWS.

Vol. LXXXIX., No. 2317.

## THE SOLUBILITY OF SOME SALTS OF THE LOWER FATTY ACIDS.

By H. STANLEY, B.Sc. (Lond.).

LUMSDEN (*Trans. Chem. Soc.*, 1902, 355) has determined values for the construction of the solubility curve for (amongst others) calcium formate. It was thought that the curves for the barium and strontium salts of the same acid might prove of interest, and possibly also that they would show similarity to a gradation from the calcium formate curve, when these elements are looked on as member of the same group in Mendeleeff's Periodic System. The calcium salts of the lower and more soluble ones of the series are noteworthy as exhibiting a decrease in solubility with rising temperature, and the barium and strontium salts might reasonably be expected to show some such deviation from the ordinary type of solubility curve, the equation to which may be given in some such form as—

$$S = a + bt,$$

where  $t$  is the temperature.

The calcium formate curve is practically a straight line, similar to those for potassium chloride, sodium nitrate, and some other inorganic salts.

Some salts of the lower fatty acids have been prepared, and their solubilities determined; below are given the results obtained for the three formates. Only three points on the calcium curve were determined, the values not showing any appreciable difference from those obtained by Lumsden.

### Preparation of the Salts.

The method adopted was the same in all cases, viz, by adding excess of the powdered carbonate to the acid, suitably diluted with water, warming, filtering, and concentrating the filtrate by evaporation to obtain the salt in crystalline form.

**Barium Formate.**—Forms monoclinic prisms, insoluble in alcohol or ether; contains no water of crystallisation.

0.9937 gm. gave 1.0027 gm.  $\text{BaSO}_4 = 59.82$  per cent Ba.

$\text{Ba}(\text{HCOO})_2$  requires 60.46 per cent Ba.

$\text{Ba}(\text{HCOO})_2 \cdot 2\text{H}_2\text{O}$  requires 56.04 per cent Ba.

**Strontium Formate.**—Forms rhombic crystals exhibiting hemihedral forms (*Pasteur, Ann. Chim. Phys.*, [3], xcxi., 98; *Jacobsen, Pogg. Ann.*, cxlii., 493), insoluble in alcohol or ether. It crystallises with two molecules of water, in this respect differing from the corresponding salts of barium and calcium. The propionate and acetate of strontium also contain more water of crystallisation than do those of barium and calcium.

0.7391 gm. gave 40.94 per cent Sr.  
 $\text{Sr}(\text{HCOO})_2 \cdot 2\text{H}_2\text{O}$  requires 41.09 per cent Sr.

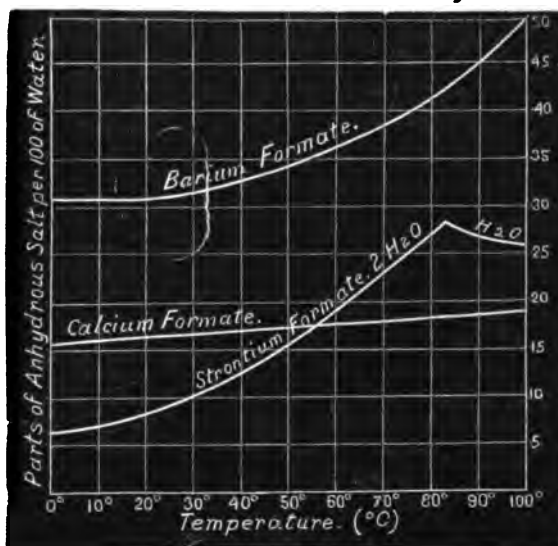
### Solubility Determination.

The solution, with excess of solid, was contained in a stout bottle, with a doubly bored rubber stopper, admitting a stirrer rotated by means of a water turbine, and a tube leading to a second vessel, into which as much solution as was required could be drawn off for analysis. A thermometer reading to tenths of a degree was used, and it was found possible with the regulator employed to keep the temperature constant, usually to one-tenth, and always to one-fifth of a degree for the requisite length of time. As the saturation point was approached, more of the solid in a finely powdered state was added, as recommended by Ostwald.

| Barium Formate, $\text{Ba}(\text{HCOO})_2$ . |                     |                             |                       |                  |                     |
|----------------------------------------------|---------------------|-----------------------------|-----------------------|------------------|---------------------|
| Temp. °C.                                    | Weight of solution. | Weight of $\text{BaSO}_4$ . | Weight of Ba formate. | Weight of water. | Percent Ba formate. |
| 0°0                                          | 1.0964              | 0.2602                      | 0.2553                | 0.8431           | 30.28               |
| 10°0                                         | 1.3951              | 0.3340                      | 0.3254                | 1.0697           | 30.41               |
| 35°0                                         | 2.1230              | 0.5333                      | 0.5195                | 1.6035           | 32.32               |
| 54°5                                         | 1.4630              | 0.4012                      | 0.3908                | 1.0722           | 36.36               |
| 73°6                                         | 2.5780              | 0.7425                      | 0.7233                | 1.8447           | 39.31               |
| 93°2                                         | 2.5503              | 0.8280                      | 0.8067                | 1.7431           | 46.25               |
| 100°0                                        | 2.8625              | 0.9310                      | 0.9070                | 1.9555           | 48.88               |

| Strontium Formate, $\text{Sr}(\text{HCOO})_2 \cdot 2\text{H}_2\text{O}$ . |                             |                       |        |        |                     |
|---------------------------------------------------------------------------|-----------------------------|-----------------------|--------|--------|---------------------|
|                                                                           | Weight of $\text{SrSO}_4$ . | Weight of Sr formate. |        |        | Percent Sr formate. |
| 0°0                                                                       | 3.8869                      | 0.3356                | 0.2550 | 3.6319 | 7.02                |
| 11°0                                                                      | 5.3809                      | 0.5309                | 0.4033 | 4.9776 | 8.08                |
| 28°6                                                                      | 1.5023                      | 0.2058                | 0.1564 | 1.3459 | 11.62               |
| 37°4                                                                      | 1.9733                      | 0.2995                | 0.2281 | 1.7452 | 13.01               |
| 51°4                                                                      | 2.1848                      | 0.4031                | 0.3036 | 1.8785 | 16.31               |
| 67°5                                                                      | 1.6596                      | 0.3735                | 0.2838 | 1.3758 | 20.62               |
| 81°5                                                                      | 3.1110                      | 0.8480                | 0.6445 | 2.4655 | 26.14               |
| 86°0                                                                      | 1.5121                      | 0.4312                | 0.3277 | 1.1888 | 27.58               |
| 91°7                                                                      | 1.6831                      | 0.4711                | 0.3581 | 1.3251 | 27.01               |
| 100°0                                                                     | 2.8097                      | 0.7762                | 0.5899 | 2.2198 | 26.57               |

| Calcium Formate, $\text{Ca}(\text{HCOO})_2$ . |    |    |    |    |                    |
|-----------------------------------------------|----|----|----|----|--------------------|
|                                               |    |    |    |    | Percent Ca formate |
| 0°0°..                                        | .. | .. | .. | .. | 16.00              |
| 51°7°..                                       | .. | .. | .. | .. | 17.32              |
| 100°0°..                                      | .. | .. | .. | .. | 18.29              |



Considerable difficulty has been experienced in getting definite values for the barium acetate curve, and the results are not yet complete. The author's best thanks are due to Mr. E. C. Martin, B.Sc., for help with this part of the work, and also to Prof. Wertheimer for granting the use of the research laboratory at the College.

Merchant Venturers' Technical College,  
Bristol.

**Directory of Paper Makers of the United Kingdom.**—We have just received the 1904 edition of this Directory, in which the list of paper makers of the United Kingdom has been carefully and authentically revised. Additions have been made to the list of trade designations used as water-marks, &c., bringing the total to over 5500; there is also a complete classification of advertisers, forming a directory of paper makers' principal engineers, purveyors of raw materials, &c., which will be of great use to those engaged in the trade. The Directory comprises 192 pages.



## THE ELEMENTS: VERIFIED AND UNVERIFIED.

By CHARLES BASKERVILLE, Ph.D., F.C.S., Smith Professor of General Chemistry, University of North Carolina.

(Continued from p. 187).

## LIST OF PROPOSED CHEMICAL ELEMENTS (continued).

| Date. | Name.                             | Source.                             | Authority.                         | Reference.                                                                                           | Remarks.                                                                                                                                                                                                                                                                                                                                                                                                                               |
|-------|-----------------------------------|-------------------------------------|------------------------------------|------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1898  | Radio-active thorium (Thorium X). | Pitchblende.                        | Curie and Schmidt (independently). | Wied. Ann., 65, 141; Mdm. Curie's thesis, Faculté des Sciences de Paris, 1903; (Chem. News, 88, 85). | See also Crookes's Thorium X and Rutherford's Thorium X. All likely same. Characteristic "emanations," perhaps due to helium (see Soddy and Rutherford, and Ramsay, Chemical News, 1903). Giesel says radio-activity not due to actinium (Berichte, 34, 3776). See Hofmann and Zerban.                                                                                                                                                 |
| 1898  | Coronium.                         | Fumarole gas.                       | Nasini, Anderlini, and Salvadori.  | Atti R. Acad. dei Lincei, Roma, [5], 7, 11, 73; Chemical News, 78, 43.                               | Spectra of gases from Solfatara di Pozzuoli and Vesuvius fumaroles showed, besides argon ( $\lambda$ 531'5) and other lines, 1474 K, attributed to the corona, hence terrestrial coronium.                                                                                                                                                                                                                                             |
| 1898  | KRYPTON.                          | Liquid air.                         | Ramsay and Travers.                | Proc. Roy. Soc., 63, 405.                                                                            | See special works on these novel constituents of the air; also Ladenberg and Krugel.                                                                                                                                                                                                                                                                                                                                                   |
| 1898  | NEON.                             | Liquid air.                         | Ramsay and Travers.                | Proc. Roy. Soc., 63, 437.                                                                            | Predicted by Johnstone Stoney from mathematical calculations; and Ramsay (Presidential Address to the Chemical Section of the British Association, Toronto Meeting, 1898).                                                                                                                                                                                                                                                             |
| 1898  | XENON.                            | Liquid air.                         | Ramsay and Travers.                | Chemical News, 78, 154.                                                                              | Very heavy gas.                                                                                                                                                                                                                                                                                                                                                                                                                        |
| 1898  | Metargon.                         | Liquid air.                         | Ramsay and Travers.                | Trans. American Assoc. Adv. Science, Boston Meeting; Chem. News, 78, 197.                            | Subsequently shown to be carbon monoxide.                                                                                                                                                                                                                                                                                                                                                                                              |
| 1898  | Etherion.                         | Air.                                | Brush.                             |                                                                                                      | Calculated atomic weight 0.001 ( $H = 1$ ). Crookes attributes the observations to watery vapour. A personal letter assures me he is still busy with the problem and "building a very large and elaborate apparatus for separating minute quantities of very light gases."—C. B.                                                                                                                                                       |
| 1898  | Polonium.                         | Pitchblende.                        | Curies.                            | Comptes Rendus, 127, 1755; 134, 85.                                                                  | Radio-active bismuth. Precipitated by hydrogen sulphide from acid solution. See also Giesel (Wied. Ann., 1899, 69, 84) and Mackwald (Berichte, 1902, 35, 2285).                                                                                                                                                                                                                                                                        |
| 1898  | RADIUM.                           | Radio-active barium in pitchblende. | Curies.                            | Comptes Rendus, 127, 1225; 129, 717; Wied. Ann., 1900, 2, 742.                                       | By prolonged fractional crystallisation of chlorides and bromides characteristic spectrum, as shown by Demarçay, Runge and Exner, and Crookes (Chemical News). For full account of this wonderful substance see Mdm. Curie's thesis, "Radio-active Substances" (Chemical News, 88, 85 <i>et seq.</i> ); Giesel, "Die Radio-activen Stoffe, &c."; Hofmann, "Radio-activity and Chemistry"; Barker; and numerous papers in the journals. |
| 1899  | Actinium.                         | Pitchblende.                        | Debiere.                           | Comptes Rendus, 129, 593; 130, 906.                                                                  | Resembles titanium in iron group. See full papers, and works of Hofmann and Zerban (Berichte, 1902, 35, 53), Baskerville (Journ. Am. Chem. Soc., 23, 761), and Brauner (Proc. Chem. Soc., 17, 67). No doubt new element. Must not be confounded with Phipson's actinium. See Uranium X.                                                                                                                                                |

|      |                                   |                                  |                       |                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
|------|-----------------------------------|----------------------------------|-----------------------|-----------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1899 | Asterium hydrogen (new). Unknown. | Stars of different temperatures. | Lockyer.              | Royal Society, February 23, 1899; Chemical News, 1899, 79, 145. | Dependent upon spectroscopy. See original paper for wave-lengths and range of stars in which the several lines may be seen. See also Pickering's "New hydrogen."                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |
| 1899 | VICTORIUM. (Monium).              | Yttria.                          | Crookes.              | Chemical News, 79, 212; 80, 49.                                 | First called "Monium." Atomic weight and spectrum criteria (also phosphorescent). Obtained by prolonged and varied fractionations of yttrium salts. Atomic weight 117.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |
| 1900 | Uranium X.                        | Uranium.                         | Crookes.              | Proc. Roy. Soc., 1900, 407.                                     | Doubtless Debierne's actinium. Hofmann and Zerban (loc. cit.) maintain minerals freed from uranium give a non-active thorium. The whole problem of radio-activity is open. Strutt secured strong radio-active gas from mercury (Phil. Mag., [6], 6, 113); Wilson showed fresh snow gives radio-active residue on evaporation (Proc. Phil. Soc. Camb., 1903, 2, 85; Geisel (Ann. Physik., 1903, 41, 10, 429), &c.                                                                                                                                                                                                                                                                                                                     |
| 1900 | Thorium α.<br>Thorium β.          | Thorium.                         | Brauner.              | Proc. Chem. Soc., London, 17, 67.                               | Hydrolysis of hepta-hydrated thorium tetra-ammonium oxalate. One with atomic weight 220; other, 260. No doubt one method, but slow at breaking thorium into its constituents. See Baskerville. Auer says quite possible thorium is not an element (Chemical News, 85, 235; Journ. f. Gas Beleucht. u. Wasservers., 1901, 661). Drossbach analysed two commercial thorium nitrates and found small amounts of different rare earths present (Zeit. f. Angew. Chem., 14, 655). He accounts for Brauner's observations by presence of ytterbium.                                                                                                                                                                                        |
| 1900 | Γ<br>Δ<br>Ω<br>Φ                  | Terbia.                          | Demarçay.             | Comptes Rendus, 131, 6; Chemical News, 82, 127.                 | Δ may be de Boisbaudran's Z <sub>7</sub> . Characteristic spectral lines. Unsettled so far.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| 1900 | Krypton II.                       | Liquid air.                      | Ladenberg and Krugel. | Akad. d. Wiss. Berlin; Chemical News, 82, 269.                  | 850 litres liquid air distilled. Molecular weight obtained = 58.81.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| 1900 | Austrum II.                       | Orthite.                         | Pribram.              | Monats., 1900, 21, 148-155; Journ. C. S., 77-8, 3, 347.         | See Linnemann. He found traces of a new element, quite distinct from Linnemann's; yet to be isolated. Independently of Brauner. Pure thorium fractionated by precipitation with sulphur dioxide, &c. Variation in atomic weights (225 for 232.6). Heavy constituent more radio-active than other, judged by photographic and electrical methods. Later work, fractioning by organic bases and distillation of chloride. Show three elements present—carolinium, true thorium, and berzelium (see). Doubtless Thorium X of Crookes and Rutherford is the radio-active element present accumulating with the carolinium. It does not necessarily follow that carolinium is radio-active, although the active body accumulates with it. |
| 1900 | Carolinium.                       | Thorium.                         | Baskerville.          | April, 1901; Journ. Am. Chem. Soc., 23, 761; Chemical News.     | Primarily active substance. They state perhaps two elements present with atomic weights 100.92 and 171.96. Atomic weight 151, characteristic spark spectrum. Phosphorescent spectrum shows it to be Crookes's S <sub>8</sub> , and reversal spectrum de Boisbaudran's Z <sub>6</sub> .                                                                                                                                                                                                                                                                                                                                                                                                                                               |
| 1900 | Radio-active lead.                | Brüggerite.                      | Hofmann and Strauss.  | Berichte, 1901, 33, 3129; 34, 907; 1902, 35, 1453; &c.          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 1901 | "Σ" EUROPIUM.                     | Samarските.                      | Demarçay.             | Comptes Rendus, 132, 1484;                                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |

(To be continued).

OBSERVATIONS ON  
SOME CONTINUOUS INTRAMOLECULAR, AND  
AT FIRST REVERSIBLE, CHANGES  
EXTENDING OVER PROLONGED PERIODS  
OF TIME.

By R. J. FRISWELL.

## PART I.

It had long been held, though until the liquefaction of the so-called permanent gases there was no experimental proof, that at a temperature approaching the absolute zero changes would be completely inhibited in bodies subjected to it, whilst, on the other hand, many years of work on the spectra of the sun and the fixed stars have proved that there is also an upper limit to such changes. Grove had arrived at the latter conclusion as far back as 1846, as a result of his work on the combination of hydrogen and oxygen by heat and electricity, and the decomposition of water by heat. (See *Phil. Trans.*, 1847, Bakerian Lecture, dated August 26, 1846, Supplement, November 10, 1846, "On certain Phenomena of Voltaic Ignition and the Decomposition of Water into its Constituent Gases by Heat." P. 15.—"If therefore there be planets whose physical condition is consistent with intense heat, the probability is that the substances that compose them are in a totally different chemical state from ours, and resolved into what we call elements, but which by intense heat may be again resolved into more subtle elements." P. 19.—

"there appears to be an extensive series of facts which afford strong hope of a generalised antagonism between thermic repulsion and chemical affinity, and a consequent establishment of the law of continuity in reference to physical and chemical attraction").

Between the very wide range of temperature here assumed, changes of all degrees of rapidity occur, and for obvious reasons investigators have concerned themselves with such as take short spaces of time. They have also almost always endeavoured to hasten them, looking more to final results than to the transition products arising during molecular interchange.

This has, I think, caused slow changes to be neglected, and has a tendency to regard substances as of far greater rigidity or stability than they really possess.

Of course much work has been done pointing in another direction, and of this none has been more remarkable than that of Roberts-Austen on the transfusion of metals.

A full realisation of the state of nature thus exhibited experimentally appears to do away with much of the apparatus, often cumbrous and unwieldy, brought in to account for changes, such as electro-chemical force, reversed electrolysis, and so on.

The energy of chemical change can be measured in terms of electrical phenomena, just as the change in the position of a body in relation to the centre of the earth can, the explanation being simply a re-statement of the doctrine of the conservation of energy.

The tendency of explanations such as the above has also been to induce a strongly held belief in the constancy of atomic constitution, or rigidity of configuration of the molecules of substances, change being looked on as arising from without the molecule.

Many years since, to account for the change of diazobenzeneanilide into aminoazobenzene, V. Meyer proposed the theory of the "labile" or movable hydrogen atom (*Ber.*, xiv., p. 2447). The view advocated attracted attention for a time, but has been somewhat overlooked amidst the growth of stereo-chemical theory.

This reaction was subsequently investigated by Friswell and Green, who showed that the cause was slight dissociation of the diazobenzeneanilide into diazobenzene and aniline (of course in the form of their salts) brought about by dilute hydrochloric acid (*Trans. Chem. Soc.* xviii., 977; xlix., 746).

Give this condition sufficient time and a favourable tem-

perature, the bodies re-arrange themselves into the molecule known as aminoazobenzene hydrochloride. It seemed, therefore, that it should be possible to produce aminoazobenzene hydrochloride directly, since the diazobenzene anilide was resolved into diazobenzene and aniline as a preliminary to its formation. But every attempt failed, and we proved that under all conditions known to us diazobenzene and aniline combined first into diazobenzene anilide, and that diazobenzene anilide then split up into diazobenzene and aniline, which very slowly combined into the aminoazobenzene molecule.

The experiments to be detailed in the second part of this paper throw a considerable light on this process, since aminoazobenzene is shown to be a base capable of decomposing aniline hydrochloride, while in the papers quoted it is shown that hydrochloric acid dissociates diazobenzene anilide into diazobenzene and aniline.

The whole of the molecules being in such a solution in a state of strain there is but one way of releasing the tension, and that is by the formation of a basic body capable of saturating hydrochloric acid.

As might be expected, there is a degradation of energy, the violently explosive diazobenzene anilide having become the stable aminoazobenzene.

The molecules have released to some extent their atoms, the system is, so to speak, in a nebular state; throw an appropriate strain on these wandering atoms, and you can compel them to assume certain configurations, by which the fact of their wandering is diagnosed, the diagnosis taking the form of an assertion of the presence of one of the complexes originally employed.

In the case before us, diazobenzene anilide is formed, and in the absence of an acid will continue to exist, the body being in fact an aniline salt, and since the diazobenzene is the more chlorous or negative portion of the molecule, it should be called aniline diazobenzide. An acid strain thrown on to it will cause its conversion into a basic body as a whole, provided the acid is one which is of so powerfully acid a character as to be able to break up the whole mass into a salt of aniline and a diazobenzene salt, and thus exclude the latter from all participation in the reaction. I would venture to suggest, in view of these results, that the character of a chemical body is a function of the strains mutually exerted between it and the reagents we make use of to "ascertain its constitution." Applying a different strain, we should assume a different constitution.

Hence there is no incongruity in bodies having two or more formulæ or playing according to circumstances two parts, as, for instance, both that of base and acid, as certain hydroxy-amino compounds certainly do.

Moreover, in the case of such bodies, the greater the acid character of the acid brought into contact with them the greater is the development of the basic power of the other member of the complex, and, conversely, the less the power of the acid the greater the acid or phenolic properties.

Thus it may be safely said that the character of the body is a function of its environment, and here again we get a difference strongly separating a chemical molecule from a rigid system.

I am therefore led to hazard the hypotheses:—

- I. That V. Meyer's "labile" or movable hydrogen atom is not the only atom endowed with variability.
- II. That "constitution" is the constitution of a substance when subjected to a particular strain.
- III. That the properties of a substance are dependent to a greater or less degree upon the body with which its properties are being explored, *e.g.*, one showing but feeble basic properties to a weak acid may, and does, develop strongly basic properties to a strong acid, and *vice versa*.

In support of the above speculations, which have arisen during a very prolonged study of the question, I beg to detail the following experiments which have extended over a long

series of years. The changes occur so slowly that months and even years may pass and still leave them incomplete.

In certain cases it will be shown that the changes can be reversed by a momentary exposure to heat, although a secondary change which always follows the first is brought about in a few hours if the heat be continued; while this change will take about two years to become perceptible, and nearly or quite four to complete if the temperature remains at or about 15° C.

(To be continued).

## NOTE ON SCALE FOR CHEMICAL VALUATION OF SOILS.

By VINCENT EDWARDS, F.C.S., F.I.C.

In a recent article on the rapid analysis of soils, I ventured to suggest a plan by which the fertility from a chemical point of view could be conveyed by a given number. At present among those who are interested in soils, there is considerable confusion of terms, vague expressions such as "great fertility," "virgin," "moderate," and so on, are freely used, and as a simple concrete expression seems to be desirable I venture to propose the following plan:—I would describe a good soil as containing 0.25 per cent of each of the three essential ingredients, nitrogen, phosphoric acid, and potash; thus, the three added together, 0.75 would equal 100, and from their analysis all soils could be in a manner compared with the "standard" imaginary one.

It might be objected that certain ill-balanced soils could not be so valued; for instance, a peat soil with excess of nitrogen. I would meet this by, as it were, only giving 25 "marks" for each ingredient. Let us suppose the peat soil contained 2 per cent nitrogen and traces of phosphoric acid and potash; this would therefore be rated at 25 on the scale for the nitrogen alone. A word of explanation, "peaty," and this figure would convey much information.

I am quite aware of the objections to this plan, but I think it does something to remedy the present confusion, and would give some information to those who have not made a study of the constituents of soil, bring them in touch with analytical chemistry, and perhaps encourage them to attempt improvements of sterile conditions with more hope of success. At present the benefits to be derived from agricultural chemistry are a sealed book to vast numbers connected with land, a most melancholy fact. I recently published a "Partial Analysis" of a garden soil containing on the day 0.12 per cent nitrogen, 0.08 per cent phosphoric acid, 0.05 per cent potash; this on the proposed scale would be 33, a figure showing at once some need of rational manurial treatment.

Laves' Works, Barking.

## A NEW PROCESS OF PREPARING VENETIAN RED.

By JOHN GEDDES MCINTOSH.

THE following new process of preparing venetian red, recently invented by me, may prove serviceable to those engaged in the industry. It simultaneously solves the question of nuisance from fumes and the admission of air into the furnace. Waste peroxide of lead can be got cheaply, and when it is furnished in molecular proportions with dehydrated ferrous sulphate, after the two have been intimately mixed, the reaction takes place according to the following equation:  $2\text{FeSO}_4 + 2\text{PbO}_2 = 2\text{PbSO}_4 + \text{Fe}_2\text{O}_3 + \text{O}$ , which corresponds to 278 parts of pure  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  (green vitriol) to 239 of lead peroxide.

When the proportions are adjusted to a nicety not a particle of fume is given off, and as there is no free acid in

the product it need not necessarily be levigated. If an excess of  $\text{PbO}_2$  be used, an equivalent amount of red lead is formed from the peroxide by the escape of oxygen. I commend this process to the notice of the alkali works inspectors as affording a solution of the problem they have been trying to solve this last ten years and more. Should enough lead peroxide not be available for manufacture on the grand scale, partial substitutes may be found in red lead and litharge; or peroxide of manganese could be used, and the resultant  $\text{MnSO}_4$  washed out from the  $\text{Fe}_2\text{O}_3$ . The  $\text{MnSO}_4$  could find a ready sale for use in dyeing, and as a raw material in the manufacture of precipitated rosinate of manganese, so erroneously termed resinate of manganese. I am aware that green vitriol has been furnished with either red lead or litharge before now by French chemists, but I do not recollect ever reading of the peroxides of lead and manganese having been used. In the case of the black oxide of manganese the presence of impurities, such as lime, need form no obstacle to the preparation of a pure sulphate of manganese. I have not thought it necessary to patent the processes, which only suggested themselves to me by a client submitting to me a sample of waste lead peroxide to see if I could find a use for it. I tried the above experiments on a large scale, and found them to produce excellent pigments.

Analytical Laboratory,  
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London, E.

## CONTRIBUTIONS TO THE CHEMISTRY OF THE RARE EARTHS.\*

LANTHANATES.

By CHARLES BASKERVILLE and GEORGE F. CATLETT.

**Synopsis.**—The resemblance of lanthanum to aluminum was taken advantage of, and the preparation of such bodies as the lanthanates and meta-lanthanates, hitherto not reported, described. The new substances are sodium tetra-lanthanate ( $\text{Na}_2\text{La}_4\text{O}_7$ ) and meta-lanthanates of sodium, potassium, lithium, and barium ( $\text{MH}_2\text{La}_2\text{O}_5$ ). Two methods were used: fusion of lanthanum oxide with alkaline carbonates, and prolonged digestion in a concentrated solution of the alkaline hydroxides at 100° C. These bodies do not serve as a means for the separation and purification of lanthanum.

Aluminum, which belongs to the third group in the periodic classification, may be separated from many elements by the solubility of its hydroxide in the non-volatile alkalis. This is due to the formation of soluble aluminates or meta-aluminates. It therefore appeared reasonable that lanthanum, which is classed with the same group, would deport itself similarly and perhaps offer a desirable new mode of separation from other or the rarer elements.

In their work on zirconates, Venable and T. Clarke proposed the following methods for the preparation of this class of bodies (*Journ. Am. Chem. Soc.*, xviii., 5; *CHEMICAL NEWS*, 1896, lxxiv., 42 and 54):—

- I. Fusing with boron trioxide (Ebelmen).
- II. Fusing with alkaline carbonates (Hjortdahl).
- III. Fusing with alkaline hydroxides.
- IV. Fusing with alkaline or earthy chlorides (Hjortdahl).
- V. Precipitation of solutions of salts with alkaline hydroxides (Watts).
- VI. Dissolving the hydroxide in strong solutions of sodium or potassium hydroxide and precipitation by dilution or by neutralisation with an acid.

\* From the *Journal of the American Chemical Society*, 1904, xxvi., No. 1.

The process used by Ebelmen was not applicable with zirconia, because that oxide does not dissolve in boron trioxide. The second and third processes given above, gave the most successful results. Water and dilute acetic acid were used to dissolve the alkali from the zirconate.

In the work on the lanthanates, those two methods, which were most successfully used by Venable and Clarke, were applied with some alterations, as will be noted in the detailed account which follows. Water was found useless as a washing medium, as, with the exception of the sodium tetra-lanthanate, the other bodies appeared to be decomposed. Acetic acid dissolved the lanthanum and supposed lanthanate with equal ease, but it was used effectively in the preparation of the lithium lanthanate. Alcohol was used in most of the experiments, and, as far as could be seen, had little action on the product. In the case of the fusion of lanthanum and barium hydroxide, a solution of ammonium chloride in water was used to dissolve out the remaining barium hydroxide.

The lanthanum compounds used were prepared from a quite pure lanthanum ammonium nitrate, generously loaned by H. S. Miner, of the Welsbach Lighting Company. The hydroxide was prepared from this compound by precipitation with ammonia, and washing until free from that volatile alkali. As is well known, lanthanum hydroxide readily absorbs carbon dioxide from the air; hence it was kept covered with water most of the time, filtered, and used fresh in each experiment. The oxide used was obtained by igniting the hydroxide obtained in platinum. Very concentrated solutions (nitrates and chloride, neutral and acid) of varying thicknesses gave no absorption bands, showing the absence of certain of the rare earths that are likely to be present.

The analyses of the preparations were made by dissolving a definite quantity of the dried body in hydrochloric acid, precipitating the lanthanum hydroxide with ammonium hydroxide. The precipitate was thoroughly washed, burned, and weighed as lanthanum oxide. The filtrate was evaporated to dryness, and the alkalis or alkaline earths converted into sulphates, and weighed as such. When water was determined, the material was ignited in combustion tubing and the moisture collected in a tared calcium chloride tube, dry air being drawn through. In several cases, either the washing medium failed to remove all of the carbonate used in the fusion, or some carbonate was formed when the material was exposed to the air. The carbon dioxide was determined in the ordinary alkali-meter by evolution and absorption.

#### EXPERIMENT I.—*Di-sodium Tetra-lanthanate*, $\text{Na}_2\text{La}_4\text{O}_7$ .

Two grms. of lanthanum oxide and 8 grms. of sodium carbonate were kept in a state of fusion for three hours. The reaction was carried out in a platinum crucible at the temperature of a blast-lamp. At the end of the fusion, it was specked with small yellowish particles, and white crystals were formed. The mass was treated with water until the wash-water was no longer alkaline, corallin being used as indicator. The white crystalline undissolved body remaining was dried at  $100^\circ$ , and, on analysis gave the following results:—

|                       |         |       |
|-----------------------|---------|-------|
| Lanthanum oxide       | .. .. . | 88.00 |
| Sodium oxide          | .. .. . | 8.00  |
| Carbon dioxide        | .. .. . | 2.50  |
| Water (by difference) | .. .. . | 1.50  |

100.00

The carbon dioxide was very probably in combination as lanthanum carbonate,  $\text{La}_2(\text{CO}_3)_3$ , as sodium carbonate would have been removed by the wash-water. Accounting for the carbon dioxide as being present in this form, we have 8.64 per cent of this carbonate or 91.46 per cent of lanthanum and sodium oxides and water. Disregarding the water and revising the calculations, we have—

|                 |         |       |
|-----------------|---------|-------|
| Lanthanum oxide | .. .. . | 90.99 |
| Sodium oxide    | .. .. . | 9.00  |

or the ratio 2 : 1, i. e.,  $\text{Na}_2\text{La}_4\text{O}_7$ , di-sodium, tetra-lanthanate, similar to the boron compound.

#### EXPERIMENT II.

##### *Hydrated Mono-sodium Poly-meta-lanthanate.*

Two grms. of lanthanum oxide and 8 grms. of sodium hydroxide were digested in a silver dish on a water-bath for thirty hours. A white mass, almost insoluble in water but decomposed by it, was obtained. Alcohol was used and the unchanged sodium hydroxide washed out; but not all of the sodium carbonate produced by the absorption of carbon dioxide from the air. The body gave on analysis:—

|                       |         |       |
|-----------------------|---------|-------|
| Lanthanum oxide       | .. .. . | 79.60 |
| Sodium oxide          | .. .. . | 2.80  |
| Carbon dioxide        | .. .. . | 3.00  |
| Water (by difference) | .. .. . | 14.6  |

Providing for the carbon dioxide as in the first experiment, we have  $\text{NaH}_9\text{La}_5\text{O}_{15} \cdot 4\text{H}_2\text{O}$ , comparable to the poly-meta-stannates in which one of the hydrogen atoms appears to have been replaced by the metal sodium.

#### EXPERIMENT III.

##### *Hydrated Mono-lithium Poly-meta-lanthanate.*

Two grms. of lanthanum oxide and eight grms. of lithium carbonate were fused in a platinum crucible for five hours. Much difficulty was experienced in washing out the alkali, which was eventually removed by careful treatment with 5 per cent acetic acid. The acid was subsequently removed by means of alcohol. A small amount of material insoluble in hydrochloric acid was found when it was examined. The analysis of the white body gave the following results:—

|                                     |         |      |
|-------------------------------------|---------|------|
| Lanthanum oxide                     | .. .. . | 82.9 |
| Lithium oxide                       | .. .. . | 1.2  |
| Carbon dioxide                      | .. .. . | 4.3  |
| Water                               | .. .. . | 9.8  |
| Body insoluble in hydrochloric acid | .. .. . | 1.3  |

99.5

Allowing carbon dioxide to be combined in the form of lanthanum carbonate, as all of the lithium carbonate was washed out with the acetic acid, we have  $\text{LiH}_9\text{La}_5\text{O}_{15} \cdot 2\text{H}_2\text{O}$  similar to the above, with the exception of having 2 molecules of water instead of 4.

#### EXPERIMENT IV.

##### *Hydrated Mono-potassium Poly-meta-lanthanate.*

Two grms. of lanthanum oxide and 16 grms. of potassium hydroxide were digested in a covered silver crucible on a water-bath for thirty hours. It was leached with alcohol, as it was found that water decomposed the material as in the case of the sodium hydroxide digestion. The white body gave, on analysis, the following results:—

|                 |         |       |
|-----------------|---------|-------|
| Lanthanum oxide | .. .. . | 79.50 |
| Potassium oxide | .. .. . | 2.53  |
| Carbon dioxide  | .. .. . | 0.40  |
| Water           | .. .. . | 18.69 |

101.12

which approximates the formula  $\text{KH}_9\text{La}_{10}\text{O}_{15} \cdot 15\text{H}_2\text{O}$ .

The body without doubt was decomposed during the washing; hence no value can be attached to the results obtained by analysis.

#### EXPERIMENT V.—*Calcium Compound.*

An attempt was made to fuse calcium carbonate with lanthanum oxide, but such great difficulty was experienced in making the fusion and removing the unchanged calcium salts that the experiments were discontinued.

#### EXPERIMENT VI.

##### *Barium Poly-meta-lanthanate*, $\text{Ba}(\text{H}_9\text{La}_5\text{O}_{15})_2$ .

Barium hydroxide was substituted for the sodium hydroxide and the digestion carried out as in the first

experiment. A concentrated water solution of ammonium chloride was used for leaching. The body obtained gave, on analysis:—

|                 |         |       |
|-----------------|---------|-------|
| Lanthanum oxide | .. .. . | 75.97 |
| Barium oxide    | .. .. . | 6.05  |
| Carbon dioxide  | .. .. . | 2.8   |
| Water           | .. .. . | 14.9  |

99.72

or, disregarding the carbon dioxide, we have  $\text{Ba}(\text{H}_2\text{La}_2\text{O}_5)_2$ . As a result of these experiments, it appears that a number of bodies, quite complex in character, may be had, which seems to show the existence of a series of compounds of lanthanum which may be termed meta-lanthanates or poly-meta-lanthanates. At the same time it is quite evident from this and other work which immediately follows, that the methods are not suitable as a means of separation of these rare earths, and really little value can be attached to the above.

A per-lanthanate, or hydrated peroxide,  $\text{La}_2\text{O}_5 \cdot x\text{H}_2\text{O}$ , was obtained by fusion with sodium peroxide. Such a body has already been reported by Melikoff and Pissarjewsky (*Zeit. Anorg. Chem.*, xxi., 70).

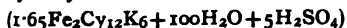
## CHEMICAL EQUILIBRIUM BETWEEN THE FERRO- AND FERRI-HYDROCYANIC ACIDS.

By MAURICE PRUD'HOMME.

If we pour, drop by drop by means of a graduated burette, a solution of chromic acid (1 part of  $\text{CrO}_3 + 200$  parts  $\text{H}_2\text{O}$ ) into a solution of yellow prussiate treated with sulphuric acid ( $2.11\text{FeCy}_6\text{K}_4 + 100\text{H}_2\text{O} + 5\text{H}_2\text{SO}_4$ ), we observe that at a certain point a drop of the mixture will turn filter-paper impregnated with tincture of guaiacol blue.

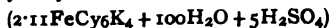
We might think that this colouration is due to an excess of chromic acid, the whole of the ferro-hydrocyanic acid having been transformed into ferri-hydrocyanic acid. In reality this is not the case, and the transformation of the ferro-hydrocyanic acid does not take place integrally. I have, in fact, made the following observations:—

### 1. Ferri-hydrocyanic acid—



alone gives a blue colour to paper impregnated with tincture of guaiacol.

### 2. Ferrihydrocyanic acid—



does not colour it.

3. The presence of small quantities of ferro-hydrocyanic acid prevents the ferri-hydrocyanic acid from turning guaiacol paper blue.

As this colouration is the result of oxidation, it must be admitted that the ferri-hydrocyanic acid is hydrolysed, giving ferro-hydrocyanic acid and oxygen. But the inverse reaction takes place, and chemical equilibrium will be represented by the two systems which change one into the other,  $\text{Fe}_2\text{Cy}_{12}\text{H}_6 + \text{H}_2\text{O} \rightleftharpoons 2\text{FeCy}_6\text{H}_4 + \text{O}$ .

The relative speeds of the two reactions would be known if we could determine the proportionate quantities of the two acids which are in equilibrium, in comparison with the reaction of the paper impregnated with tincture of guaiacol. Ordinary filter-paper will do very well for this purpose.

1. Ordinary filter-paper is coloured blue almost immediately when a drop of ferro-hydrocyanic acid of the above mentioned strength is placed on it. This property is evidently due to defective washing during manufacture after chlorination. The ferro-hydrocyanic acid, which gives prussian blue on oxidation, is oxidised by the paper itself.

2. Ferri-hydrocyanic acid, at the above mentioned strength, makes a yellow stain on this paper; this turns slightly green after a fairly long interval.

3. A mixture of ferro- and ferri-hydrocyanic acids makes a yellow stain on filter-paper, becoming green rapidly, and the more of the former acid there is in the mixture the quicker the green colour appears.

With known volumes of ferro-hydrocyanic acid solution, we note the volumes of chromic acid solution necessary to cause the appearance of the blue colouration on paper impregnated with tincture of guaiacol. Then we continue to add chromic acid, using ordinary filter-paper for the reaction, until a drop of the mixture causes a permanent yellow colouration. The ratio between the two amounts of chromic acid thus measured was found to be constant and equal to 10:1. This figure represents the ratio of the speeds with which the two inverse reactions take place.

*Estimation of the Yellow Prussiate.*—We can estimate the yellow prussiate by utilising the properties just described above. Its aqueous solution is treated with a suitable amount of sulphuric acid, about 5 per cent, and then with a 5 per cent solution of chromic acid. The blue colouration of the paper impregnated with guaiacol shows that the end of the reaction is approaching. At this moment we substitute ordinary filter-paper, which takes successively a green colouration, becoming more and more yellow.

We stop when the stain remains a pure yellow. With the above mentioned strengths it is necessary to deduct from the number of c.c. of chromic acid a constant volume of 0.25 c.c. added in excess to obtain the persistent yellow colouration.—*Bull. Soc. Chim.*, Series 3, vol., xxix., No. 20.

## EXAMINATION OF SOME ANCIENT SAMPLES OF BREAD.

By L. LINDET.

THE fragments of bread, carefully collected and preserved by archaeologists, have an appearance and chemical composition which depends on the physical conditions to which they have been exposed. I thought it would be of interest, as I have a certain number of samples in my possession, of which several were collected by Aimé Girard, to examine the whole question.

1. *Bread from Pompeii.* (Sent to A. Girard by Prof. Cannizzaro, in the name of the Minister of Education in Italy).—The breads found at Pompeii in 1862 are the best known; they have been described and analysed by de Luca (*Comptes Rendus*, 1863, vol. lvii., p. 475); they have the appearance of a porous carbon in which no trace of any of the elements of bread can be found, and they contain, in the same manner as coke or animal black, a considerable proportion of nitrogen, varying from 2.6 to 2.8 per cent. This nitrogen is not in the form of ammoniacal salts, susceptible of being displaced by magnesia, nor in the form of amines decomposable by soda. It is in the particular form which can be best described as cyanic nitrogen, capable of giving indol, pyridine, or paracyanogen by the action of dry heat; the nitrated molecule exists, in fact, in the last stage of its decomposition. It is evident that this amount of nitrogen remaining in the carbon of a calcined organic substance depends on the temperature to which this substance has been subjected; so that the estimation of the nitrogen gives us some idea of the temperature to which these samples of bread have been exposed. By heating a piece of compact bread, similar to that which would most likely be made at the time, to 350–400° in a closed vessel. I obtained a porous carbon identical in appearance with those samples found at Pompeii, containing 2.81 per cent of nitrogen, while one of the specimens now in my possession contains 2.65 per cent. Geologists agree in stating that the ashes of Vesuvius,

\* To estimate this temperature, I placed a few pieces of lead (fusing at 325°) and zinc (fusing at 412°) in the bread; the former only were fused.

which overwhelmed Pompeii in the year 79 B.C., were not very hot.

All traces of starch and cellulose have disappeared; hydrolysis does not give any reducing sugary material; however, some ulmic materials remain, capable of giving a small quantity of acetic acid on dry distillation.

The crumbs give the reaction for chlorides very distinctly; it is known that the Romans put salt into their pastry (Pliny, "*Farina subigitur priusquam addatur sal*," "*Hist. Nat.*," Book XVIII., p. 26).

II. *Bread from Lake Dwellings.*—It is in a similar condition that the specimens of bread from lake dwellings were found, discovered in different lakes after the burning of the dwellings placed therein.

In a sample from Corcellettes in Lake Neuchâtel (the bronze age), I only found a nitrated carbon ( $N = 2.46$  per cent) enclosed in vegetable *débris*, dredged up from the bottom of the lake. In another sample, less carbonised and of a reddish-brown colour, found in Lake Bourget ( $N = 4.69$  per cent), I was able distinctly to distinguish the *débris* of grains still existing, and especially fragments of the outer skin of the grain of barley, as well as of grains of starch. Further, barley is the oldest cereal known; four kinds of barley have been found in the districts of Switzerland and the Savoy (De Candolle, "*Origin of Cultivated Plants*," p. 296).

III. *Bread from Egyptian Tombs.*—Bread intended for the use of the dead, and which was enclosed in their tombs, has been admirably preserved, and is found to-day in the same condition as in which it was enclosed. In Egyptian tombs, we meet sometimes with unleavened bread in the form of flat cakes, and sometimes with leavened bread. Further, the Egyptians were familiar with yeast, since the Hebrews made use of it. The two samples I have had (sent by M. Nachet) were of unleavened bread, and entire, in which I could easily recognise the *débris* of the husk of the barley (outer epidermis, fibrous hypoderm of the grain, cells of proteic acids, radicular hairs). Now we know that the gluten of barley has no elastic properties; barley bread will not rise. The paste contains a quantity of gluten and a quantity of starch, which may be considered as normal. In my two samples I found 11.25 and 11.40 per cent of nitrogenous material ( $N = 1.80$  and 1.83), and 68.0 and 65.2 per cent of starch. This starch, as in a baked bread, is found in two forms; one part is solubilised in the form of soluble starch and dextrin (20.4 per cent). It is of more importance than in the ordinary cases, because this bread, slightly browned, has evidently been heated to a high temperature, as is proved by the acidity of the crust. The other part is in the form of swelled-up crumb; only a very few grains of starch have remained intact.

I have shown (*Comptes Rendus*, 1902, vol. cxxxiv., p. 908) that the temperature might have been estimated from the amount of gelatinisation of the starch, by treating the bread with hydrochloric pepsin (1.5 c.c. of HCl), and there again we observe that the amount of starch dissolved in the dilute acid goes beyond the ordinary limits (21.3 per cent). One of these samples of bread contains 4.5 per cent of ash, in which the presence of chlorides is found. The salt used by the Egyptians was rich in nitrates; these latter can be easily detected in the bread by means of diphenylamine.

IV. *Roman Bread from Aosta.*—In 1856, researches brought to light at Aosta, in a place called La Maria, various Roman objects, among which was found a fragment of bread, representing the quarter of a round loaf 30 or 40 c.m. in diameter. This loaf was entirely transformed, by decay and refilling of the matrix, into a morsel of sandstone consisting of granitic elements (mica and felspar). This pseudomorph has an interest that is not entirely geological. I have found, in fact, several grains of starch embedded in the mineral matter, and this leaves no doubt as to the nature of the fragment discovered at Aosta. These grains of starch, not broken down by cooking, have resisted the action of the water which

brought the granitic elements; the grains of corn which were in the mucous condition have been destroyed, as was also the gluten. Microscopic examination leads me to the conclusion that these grains of starch are from wheat.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 23.

## PROCEEDINGS OF SOCIETIES.

### THE FARADAY SOCIETY.

Ordinary Meeting, April 13th, 1904.

THE meeting was held at the Institution of Electrical Engineers, Mr. J. SWINBURNE, Vice-President, in the Chair.

Mr. ARTHUR J. HIGGINS read an abstract of his paper on "*Alloys of Copper and Arsenic*," illustrating his remarks by a series of interesting and beautiful lantern slides.

The object of the author's investigations was to ascertain the exact relation between copper and arsenic in binary alloys, and the limit of proportion of arsenic that can be retained in copper in the cold solid state.

The addition of arsenic lowers the melting-point of copper uniformly down to about 14 per cent, when a steep fall in the freezing-point curve occurs, reaching its lowest point at 68.5° C. This alloy contains 19.2 per cent of arsenic, which corresponds to the formula  $Cu_2As_2$ . The alloy with 22 per cent of arsenic freezes at 70.8°, and the temperature gradually rises until the alloy with 28.34 is reached at 74.7°. This is the compound  $Cu_3As$ . At 81.0° another chemical compound freezes, having the chemical formula  $Cu_5As_2$ ; it contains 32.2 per cent of arsenic. Beyond this point the temperature gradually falls again to a minimum at the alloy, with about 35 per cent of arsenic. The curve then rises to another summit at 74.0°, forming the compound  $Cu_2As$ , with 37.24 per cent of arsenic. From this position the curve descends to 70.2° with the alloy containing 41 per cent of arsenic; this is nearly the practical limit of the direct combination of copper and arsenic.

The top surfaces of many of the alloys were photographed. The types of pattern visible on the etched surface depend upon the position of the freezing-point on the curve. For alloys whose freezing-point is on or near the summit of a curve, the whole of the section is composed of one substance, generally made up of crystal grains having fine boundaries. When by addition of copper or arsenic a summit of a curve is left, the lines between the crystal grains widen and become filled with a network of matter differing from that of the crystals, and generally showing a more or less striated appearance, so characteristic of eutectics. As the bottom of a curve leading from a summit is reached, the eutectic occupies the whole surface. It is noticeable that the alloy of minimum fusion of copper-arsenic alloys has an extremely fine striated structure. Any deviation in composition from the eutectic proportion causes the breaking up of this striated pattern, and grains of one of the constituents appear in the eutectic matrix.

A microscopic examination was also made of polished sections of the various alloys. These when viewed in daylight are all copper-coloured in those with little arsenic, and gradually get paler as the arsenic is increased. When the alloy with 19.2 per cent is reached, the surface appears of a pale blue colour, and this is continued up to the compound  $Cu_3As$ , containing 28.34 per cent, which is of a deeper blue colour. The alloys between 19.2 and 28.3 per cent have a paler blue tint, because the blue constituent is associated with a constituent richer in copper, forming a eutectic mixture. The alloy with 30.0 per cent is of a light purple colour, and when the compound  $Cu_5As_2$  is reached, containing 32.2 per cent, a full reddish purple is obtained. This purple colour maintains up to the limit of



these experiments with 45 per cent of arsenic, but gets gradually paler.

The paper concludes with a detailed description of the various alloys, fully illustrated by excellent photomicrographs.

Mr. SWINBURNE, referring to the melting-point of arsenic, which the author thought could only be obtained by experiments made under great pressure, observed that the effect of pressure on the melting-point of solids is very small, and that probably at ordinary pressures the melting-point of arsenic was very close to its volatilising point.

Dr. PERKIN hoped that the author would supplement his researches by making conductivity measurements of the series of alloys.

Dr. C. H. DESCH made some interesting comparisons between the results obtained by the author and those published by other investigators, in the case of the analogous series of copper-antimony alloys.

Mr. E. G. P. BOUSFIELD contributed a paper on "*Experiments with a New Primary Cell.*"

The cell consists of an inner porous pot containing nitric acid and a carbon pole, and an outer vessel containing sodium hydrate solution and a metal pole, preferably zinc, *i.e.*, a solution of from 12 per cent to 15 per cent, using solutions of maximum conductivity with zinc and carbon poles on open circuit, an E.M.F. of 2.6 volts may be obtained. Not only does the cell possess this comparatively high E.M.F., but it may be short-circuited far longer than most cells before it runs down. A cell short-circuited through a total resistance of 0.61 ohms gave a current of 4.18 amperes, which fell to 2.61 in an hour, 2.38 in two and a quarter hours, and 1.75 in six hours. A smaller cell gave a fairly constant current of about 0.8 ampere for twenty or twenty-five hours. Discharge curves are given in the paper.

Acids other than nitrate were experimented with. Some of them gave higher E.M.F.'s than nitric acid, but no other acid possessed the same "lasting powers." The effect of using various metals instead of zinc was also tried; some of these gave higher E.M.F.'s than the zinc, but the high readings were quite transitory. Using two carbon poles the E.M.F. was about 1.35 volts. The cell is then similar to Becquerel's platinum cell, in which the plates are immersed in acid and caustic potash respectively.

Mr. W. R. COOPER referred to the disadvantages of both nitric acid and caustic soda as electrolytes. The high E.M.F. of the cell was only apparent, because it rapidly fell to a working value of 1.8 or 1.9. He would like to have seen comparative tests made, say with a bichromate cell.

Mr. J. SWINBURNE mentioned a suggestion made by Dr. Swan, that the cell would be improved if it were converted into a three-fluid cell, with a neutral electrolyte (*e.g.*,  $\text{NaNO}_3$ ) in the mid compartment.

Mr. BOUSFIELD also contributed a "*Note on Determining Accurately the Percentage of Ozone in Gases not Dissociated by Moderate Heat.*"

Difficulty was found in determining accurately the quantities of ozone formed in different electrolytic reactions. The potassium iodide test is sufficiently accurate for most purposes, but for very accurate work it is not satisfactory, as an error of 5 per cent or more is often obtained with a slightly alkaline solution, and an error of as much as 1 or 2 per cent with an acid solution. Estimating the ozone by weight, using glass globes for that purpose, was also found to be somewhat uncertain, due to the condensation of moisture on the glass. The method recommended by the author is a measurement by volume. A certain quantity of gas containing ozone is measured. This gas is then passed over some substance, such as heated platinised asbestos, which is capable of converting ozone into ordinary oxygen. On again measuring the gases, an increase of volume due to the breaking down of " $\text{O}_3$ " molecules into " $\text{O}_2$ " molecules is

found. From this increase of volume the quantity and percentage of ozone originally present is readily calculated. A description of the simple apparatus required for the measurements is given in the paper.

## NOTICES OF BOOKS.

*Electrolytic Preparations.* By Dr. KARL ELBS. Translated by R. S. HUTTON, M.Sc. London: Edward Arnold. 1903. Pp. 100.

THIS volume consists principally of exercises for use in the laboratory by chemists and electro-chemists, a branch of the subject which has not hitherto been very fully dealt with in text-books. It is assumed that those who intend to make use of these exercises are already familiar with practical chemistry and physics, and especially the laws governing electric currents.

The first sixteen pages are on general matters connected with the source of current, connections, resistances, measuring, and other apparatus to be used.

Part II., or the "special" part, fills the rest of the book, and is divided into various sections and sub-divisions.

First in order come examples from inorganic chemistry with insoluble anodes, then others with soluble anodes.

The section on organic chemistry includes the electrolysis of organic acids, electro-chemical methods of reduction, and electro-chemical oxidation methods.

A careful study of this volume will put into the hands of the chemist another means for the preparation of a large number of chemical substances, and will familiarise him with the growing subject of electrolytic analysis.

*Aids to Chemistry.* By T. A. HENRY, D.Sc.(Lond.), F.C.S., &c. Eighth Edition. London: Baillière, Tindall, and Cox. 1903. Pp. 316.

IN this edition of "*Aids to Chemistry*" it has been found necessary to entirely re-write it, and to assist the medical and pharmaceutical student as much as possible, the theoretical portions of the book have been treated on broad lines, and, as far as possible, in immediate conjunction with experimentally ascertained facts serving to illustrate them.

The first portion of the book deals with inorganic chemistry, the properties and characteristics of the metals and their salts, while the latter part is on organic chemistry, or the chemistry of the carbon compounds. The whole is well arranged and adapted for the use of students.

*The Phase Rule and its Applications.* By ALEX. FINDLAY, M.A., Ph.D., D.Sc. London, New York, and Bombay: Longmans, Green, and Co. 1904.

THE first of the proposed series of manuals dealing with different branches of physical chemistry is devoted to the study of the principles underlying the phase rule of Willard Gibbs. The treatment of the subject is quite unmathematical, and, comparatively speaking, elementary. The least satisfactory part of the book is certainly that which treats of the general exposition of the rule, which requires very close attention throughout, and would probably be in places rather beyond the comprehension of the young student. The application of the general principles to the study of various examples is, however, in all respects excellent; as, for example, in the chapter giving a general summary of the various relationships discovered on applying the rule to systems of one component. Under the heading of two components are studied the phenomena of dissociation and solution; the treatment of three and four component systems, though very lucid, has been necessarily somewhat restricted owing to the great complexity of these systems.

With the volume is bound the "General Introduction to the Study of Physical Chemistry," written by Sir William Ramsay, the editor of the series.

## CORRESPONDENCE.

### THERMO-CHEMISTRY OF ELECTROLYTIC DISSOCIATION.

To the Editor of the Chemical News.

SIR,—It is with pleasure I have read Mr. P. J. Beveridge's letter (CHEMICAL NEWS, lxxxix., p. 82), in which he has taken exception to the calculation used by Mr. Richards (CHEMICAL NEWS, lxxxix., p. 31, 37) in support of his theory of solution. Mr. Beveridge has clearly pointed out the remarkable coincidence that the concentration in the single example cited from the work of Thomsen is the only one which, applying a calculation for outer work as Mr. Richards has done, gives an agreement with the observed heat of neutralisation, 13,740 calories.

Mr. Richards's paper, read at general meeting of the American Electro-chemical Society, Niagara Falls, Sept., 1903, happened to be brought into the discussion at the meeting of the New York section of that society, January 26th, 1904. As this numerical agreement of Prof. Richards was mentioned, the writer called the attention of the Section to the fact that he, in calculating this external work, had been unable to obtain this agreement even in the example given by Richards. And that this seemed due to an erroneous application by Prof. Richards of the ordinary gas laws.

The example quoted gives the reaction of two molecules, NaOH and 2HCl, in 800 water, with liberation of 27,480 calories heat of neutralisation, or 13,740 for each molecule. 800 H<sub>2</sub>O having a volume of 14.4 litres, and the osmotic pressure of the OH' and H' being, according to Richards, 6.19 atmospheres, the volume energy is  $6.19 \times 14.4$  litre-atmospheres.

The thermodynamic constant,  $2T$ , is the value in calories for RT in the equation  $pV = RT$  when  $p = 1$  atmosphere,  $v = 22.4$  litres (volume of 1 molecule gas at 0°C. at atmospheric pressure). Nevertheless, Prof. Richards in his example, having a volume smaller than the normal and a pressure greater, took into account only the ratio of the pressure to the normal pressure, and not, as he should have, the ratio of the product of the pressure and volume to the product of the normal pressure and volume, which would give the following form of calculation:—

$$\frac{6.19 \times 14.4}{1 \times 22.4} \times 2T = 4 \times 2T = 8T \text{ calories.}$$

That as Prof. Richards distinctly states this is the external work of evaporating two molecules H<sub>2</sub>O, and as the 10,110 calories latent heat to which it is added is just as distinctly stated by him to be for one molecule H<sub>2</sub>O ("18 parts"), the above external work should be divided by 2, which gives  $2(2T)$  cal. = 1164 calories,  $10,110 + 1164 = 11,274$  calories. The difference between which and 13,740 is 2466 calories, and not 40 as he has obtained. The very form of the gas law,  $pV = RT$ , teaches us that since the product of pressure and volume for any given temperature is constant, the external work necessary to form a molecule of gas is a constant quantity independent of the pressure under which the gas is formed. The work then to form one molecule any gas, no matter what the pressure is, and can only be  $2T$  calories.

On the other hand, for substances in solution which dissociate, and therefore which show an osmotic pressure greater than they would were they gaseous and occupying similar volume, the van't Hoff equation,  $pV = iRT$ , holds, and since if in the above aqueous solution we assume complete dissociation (as Mr. Richards must have done to obtain calculated osmotic pressure of 6.19 atmospheres),  $i = 2$ . The work needed to force one molecule

H<sub>2</sub>O into the two ions, OH' and H', is given directly by  $iRT = 2 \times 2T = 4T$  calories. So Richards's calculation is unnecessary, as it is merely a reversal of that made by him in obtaining the osmotic pressure in the first place.

Thus, then,  $10,110 + 1164 = 11,074$  calories out of 18,740 is the utmost that Prof. Richards's theory can account for on a basis of mere physical change, and as Prof. Richards's whole theory is to the fact that in diluted solution the elements are in an ideal combined condition, and that therefore no further chemical combination can occur, it is hardly necessary to point out that not only has Prof. Richards's failed in substantiating his theory, but that the figures given by him are rather a strong argument that the converse is true.

In the second example, since the heat for the formation of the gas is independent of the pressure, the calculation of  $(619 - 1) \times 2T = 3021$  calories is without meaning, and that therefore he has proved nothing, or rather proven that the "ionised water molecule" is not identical with the gaseous state of water-vapour at the same pressure.

The following result can, however, be obtained from the values for H' and OH' given by Richards from his tables.

Since  $\frac{1}{2}(H_2) = H' - 900$  calories, and  $\frac{1}{2}(O_2) + \frac{1}{2}(H_2) = OH' + 56,100$  calories, and H' and OH' cannot continue their separate existences in the same solution, but react with further liberation of 13,740 calories, we have the equations  $\frac{1}{2}(H_2) + \frac{1}{2}(O_2) + \frac{1}{2}(H_2) = H' + OH' + 55,200$  cal.,  $H' + OH' = H_2O + 13,740$  cal., then  $(H_2) + \frac{1}{2}(O_2) = H_2O + 55,200 + 13,740 = H_2O + 68,940$  calories.

This is for the formation of 1 molecule liquid water in diluted solution. The evaporation of this 1 molecule H<sub>2</sub>O will require 10,692 calories, leaving 58,252 calories, which number does agree with Regnault's 58,100 calories for the direct formation of water-vapour from oxygen and hydrogen gases. However, in this we have taken into account 13,740 calories chemical action which does occur and which Richards has ignored.

The elaborate tables of thermo-chemical constants which Richards has prepared in no wise help his theory, since they are but an elaboration of, and another name for, the heats of ionisation, a short table of which was published by Ostwald in 1893 (*Zeit. für Physik. Chem.*, xi., p. 501). These as far as they are given agree with Richards, but differ from Ostwald's re-calculated values given by him in his "Grundriss der Allgemeinen Chemie," p. 281.—I am, &c.,

H. A. JACKSON.

March 15th, 1904.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 12, March 21, 1904.

**Density of Fluorine.**—Henri Moissan.—After the discovery that fluorine, when absolutely free from hydrofluoric acid, does not act on dry clean glass, a series of new determinations of the density of fluorine are made by filling a glass bulb with the gas and weighing. The author describes the various precautions necessary for these determinations. His four experiments give respectively the numbers 1.298, 1.319, 1.313, and 1.312; the mean is 1.31. The theoretical density should be  $0.06927 \times 19.05 = 1.319$ , taking 19.05 as the atomic weight of fluorine. These two numbers are almost identical, and 1.31 can therefore be admitted as the experimental density of fluorine.

**Colloidal Solutions. Application of the Rule of Phases to an Investigation of the Precipitation of Colloids.**—Victor Henri and André Mayer.—The rule of phases, when applied to colloidal solutions in the same manner as to all normal solutions, allows of an examina-

tion and systematic classification of the conditions of precipitation of the whole series of colloids.

**Transformation of Oxides and Oxygen Salts into Chlorides.**—C. Matignon and F. Bourion.—The action of sulphur chloride and chlorine allows of the transformation of oxides and oxygen salts into anhydrous chlorides. This transformation can in many cases be advantageously employed for analytical work.

**Silver and Lead Salts of Monoalcoylphosphoric Acids.**—J. Cavalier.—Monoethyl- and monoallyl-phosphoric acids behave towards silver and lead salts in an analogous manner to phosphoric acid. The soluble mono-metallic salts, neutral to methyl-orange, give, on addition of lead or silver nitrate, a precipitate of bi-metallic salt, and the liquor becomes acid to methyl-orange. The mono-metallic salts of lead and silver can only be formed in a very strongly acid medium.

**Certain Amino-alcohols of Tertiary Alcoholic Function of the Type**  $R-\overset{\text{CH}_3}{\underset{\text{CH}_2\text{N}<\overset{\text{CH}_3}{\text{CH}_3}}{\text{COH}}}$  — E.

Fourneau.—The author obtains the amino-alcohols of tertiary alcoholic function by heating the chlorohydrines with a little more than two molecules of a tertiary or secondary amine dissolved in alcohol. These are isolated by driving out a portion of the solvent by boiling; this contains the excess of amine. He then separates the base by dilute hydrochloric acid. This base is freed again by caustic soda, after carefully washing the acid solution with benzene. The base extracted by ether is distilled *in vacuo*, and the yield is 80 per cent.

## MISCELLANEOUS.

**Research on Dicalcic Phosphate. Artificial Production of Brushite and Monettite.**—A. de Schulten.—The hydrated dicalcic phosphate,  $\text{PO}_4\text{CaH} + 2\text{H}_2\text{O}$  (brushite), is easily obtained in microscopic crystals when we leave the salt precipitated from  $\text{CaCl}_2$  by  $\text{PO}_4\text{Na}_2\text{H}$  in contact with its mother-liquor. The author has obtained these crystals of a measurable size by dissolving  $\text{PO}_4\text{CaH}$  in the cold, up to saturation-point, in 25 per cent acetic acid, then letting the filtrate evaporate slowly in the cold. Wash with glacial acetic acid, alcohol, and ether. Clinorhombic tables are formed, flattened along  $g_1$  with  $pk_{205}(d_1b_1g_1)$ , isomorphous with pharmacolite,  $\text{AsO}_4\text{CaH} + 2\text{H}_2\text{O}$ ;  $D = 2.317$ . The author has attempted to obtain the lower hydrates at a higher temperature (metabrushite which should be  $1.5\text{H}_2\text{O}$  is very doubtful). Starting at  $50^\circ$ , and especially at about  $100^\circ$ , the brushite is transformed into the anhydrous salt,  $\text{PO}_4\text{CaH}$  (monettite). A solution of  $\text{CO}_3\text{Ca}$  in  $\text{PO}_4\text{H}_3$ , filtered and heated to  $155^\circ$  (and even to  $260^\circ$ ), gives anorthic crystals, generally flattened along  $h_1$ , with the facets  $pg_1mo_1c_1(c_1b_1h_1)$ .—*Bull. Soc. Min.*, vol. xxvi., p. 11.

**The Use of Hydroxylamine and Hydrazine in Qualitative Analysis.**—E. Kiroevenagel and E. Ebler.—The salts of hydroxylamine or of hydrazine may be used for the separation of the metals precipitated by sulphuretted hydrogen. The principle of the method is as follows:—The precipitated sulphides are well washed and dissolved in aqua regia, the excess of acid is evaporated off, and the residue taken up with water, and precipitated by a mixture of NaOH and a solution of sulphate of hydrazine or hydrochlorate of hydroxylamine; after boiling, allow to cool, and filter. The precipitate ( $P_1$ ) contains Hg, Cu, Cd(OH)<sub>2</sub>, Bi(OH)<sub>3</sub>, and traces of Ag; the filtrate ( $F_1$ ) contains  $\text{AsO}_4\text{Na}_3$ ,  $\text{SbO}_4\text{Na}_3$ ,  $\text{Na}_2\text{SbO}_3$ ,  $\text{Pb(ONa)}_2$ , and traces of bismuth. The precipitate ( $P_1$ ) is dissolved in nitric acid, and the solution, on the addition of ammonia, throws down Bi(OH)<sub>3</sub>; the filtrate is treated with hydrochlorate of hydroxylamine, which precipitates the Hg; if copper is present the solution is decolourised while main-

taining the copper in solution; it is precipitated by means of sulphocyanide; nothing remains in solution but the cadmium which is precipitated by sulphuretted hydrogen. The filtrate ( $F_1$ ) is saturated with  $\text{H}_2\text{S}$ , the lead separates out, there remain the tin, arsenic, and antimony. On the addition of dilute sulphuric acid, the sulphides of these metals are deposited, and can be separated in the usual manner.—*Berichte*, vol. xxxv., p. 3055.

**The Formation of Peroxides in the case of Iron.**—M. Manchot.—The principle of these experiments is as follows:—A ferrous salt is submitted to the action of an oxidising agent simultaneously with an acceptor. The acceptor is a body whose oxidation, very slow if it was the only reducing agent, is made very rapid by the simultaneous presence of the ferrous reducing agent. Under these conditions we observe both the oxidation of the ferrous salt and of the acceptor. If the latter is in excess, the oxygen that it uses up, added to that which has changed the ferrous salt to the state of ferric salt, gives the total oxygen taking part in the reaction, and furnishes the formula of the transitory peroxide which is formed. This oxide comes directly before the ferric degree. Using chromic acid as the oxidiser and hydriodic acid as the acceptor, we find that the primary oxide is  $\text{Fe}_2\text{O}_5$ . The same is the case with permanganate and tartaric acid, and with  $\text{H}_2\text{O}_2$ . Oxygen gas gives  $\text{FeO}_2$  as the primary oxide. Hypochlorous acid gives  $\text{FeO}_3$ . The ferric salts themselves are distinctly capable of peroxidation. Thus their presence accelerates the decolouration of indigo by oxidising agents (less decidedly, it is true, than with the ferrous salts). The oxidising action of dilute peroxide of hydrogen is prevented by the presence of acids. The oxides  $\text{FeO}_2$  and  $\text{FeO}_3$  have not been isolated, but their existence as primary oxides does not seem the less proved by these experiments.—*Liebig's Ann. Ch.*, vol. cccxv., p. 105.

## MEETINGS FOR THE WEEK.

MONDAY, 25th.—Society of Arts, 4.30. (Cantor Lectures). "The Majolica and Glazed Earthenware of Tuscany," by Prof. R. Langton Douglas, M.A.  
TUESDAY, 26th.—Royal Institution, 5. "The Transformations of Animals," by Prof. L. C. Miall, F.R.S.  
WEDNESDAY, 27th.—Society of Arts, 8. "The Need of Duty-free Spirit," by Thomas Tyrer.  
THURSDAY, 28th.—Royal Institution, 5. "Dissociation," by Prof. Dewar, F.R.S., &c.  
FRIDAY, 29th.—Royal Institution, 9. "Westminster Abbey in the Early Part of the Seventeenth Century," by The Very Rev. J. A. Robinson, D.D.  
SATURDAY, 30th.—Royal Institution, 3. "Jewellery," by Cyril Davenport, F.S.A.

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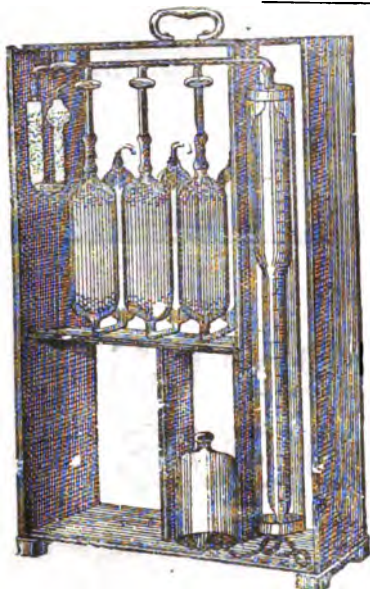
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### CONTENTS.

| ARTICLES:—                                                                                                                                   | PAGE |
|----------------------------------------------------------------------------------------------------------------------------------------------|------|
| Physical Constants at Low Temperatures, by Prof. James Dewar, M.A., F.R.S., &c.                                                              | 205  |
| Observations on some Continuous Intramolecular, and at first Reversible, Changes extending over Prolonged Periods of Time, by R. J. Friswell | 207  |
| The Elements—Verified and Unverified, by Charles Baskerville, Ph.D., F.C.S.                                                                  | 210  |
| London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.                                                                 | 211  |
| Hausmannites from Sweden, by A. Gorgeu                                                                                                       | 211  |
| PROCEEDINGS OF SOCIETIES—                                                                                                                    |      |
| PHYSICAL SOCIETY                                                                                                                             | 212  |
| NOTICES OF BOOKS                                                                                                                             | 213  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                        | 215  |
| MISCELLANEOUS                                                                                                                                | 215  |
| MEETINGS FOR THE WEEK                                                                                                                        | 215  |

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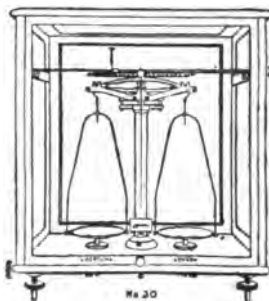


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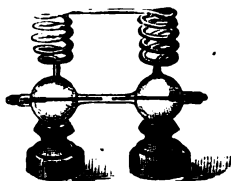
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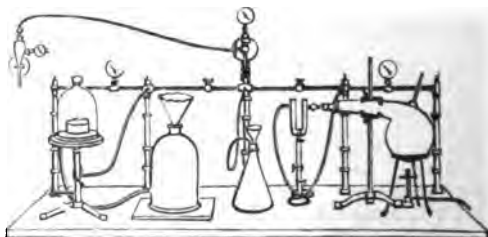
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2318.

## PHYSICAL CONSTANTS AT LOW TEMPERATURES.\*

### I. THE DENSITIES OF SOLID OXYGEN, NITROGEN, HYDROGEN, &c.

By Prof. JAMES DEWAR, M.A., LL.D., D.Sc., F.R.S.

1. The following experiments on the solid densities of oxygen, nitrogen, and hydrogen were carried out as part of a former investigation dealing with gaseous densities at low temperatures ("The Specific Volumes of Oxygen and Nitrogen Vapour at the Boiling-point of Oxygen," *Roy. Soc. Proc.*, vol. lxxix.).

The method adopted was to measure the volumes of the gases sucked into a cooled space of known capacity, when the temperature was such as in the first place to induce liquefaction, and finally solidification. For such experiments to be successful the rate of liquefaction and the cooling must be under thorough control, otherwise the cooled space may not get completely filled with solid. Further, the volume of gas condensed ought to be as large as possible, in any case about 20 litres, in order to diminish errors inseparable from the mode of manipulation. The inertia of the bell-jars of the large gas-holders causes some variation in the pressure; and errors of their calibration, and want of uniformity of temperature in the mass of gas, are all important factors. As my object was to ascertain experimentally the limiting density in the solid state, the elimination of these variations was not so important as it would have been for the study of fluid density.

The dry purified gas was contained in a gas-holder connected by a pipe with a glass bulb of 20 or 30 c.c. capacity, sealed to a narrow tube some 10 c.m. long with a glass stop-cock at the end. The glass bulb was immersed in liquid oxygen, nitrogen, or hydrogen contained in a vacuum-jacketed vessel, with arrangements for lowering the temperature of the liquids by exhaustion. The narrow tube just above the glass bulb had a mark engraved upon it, and the volume up to this point, which is to be filled with liquid or solid after cooling, was antecedently carefully calibrated. The glass stop-cock on the projecting part of the glass tube outside the vacuum vessel enabled the rate of the gas supply to be under complete control. The real difficulties were those of manipulation.

The temperatures employed were the boiling-point of oxygen, 90.5°; the boiling-point of nitrogen, 77.5°; the melting-point of nitrogen, 62.5°; the boiling-point of hydrogen, 20.5°; hydrogen boiling under 76 m.m. of pressure taken as 14.7°; and hydrogen (solid) under 10 m.m. of pressure taken as 13.1°.

Allowance for the contraction of the bulb was made by taking 0.0000250 as the coefficient of contraction (cubical) of the glass. It is possible that in going to very low temperatures (below -200° C.), this coefficient ought to be less, such as 0.0000225. An estimate made of the difference between the two would come within the range of errors, consequently the former value which was adopted in the earlier investigations was retained.

The weight of 1 litre of oxygen at 0° C. under 760 m.m. pressure was taken as 1.430 grms., of nitrogen as 1.2564 grms., and of hydrogen as 0.0899 grm.

The observations and results are given in the following tables:—

TABLE I.—Oxygen.

| No. | Description.    | T'.    | V.      | T.    | p.    | d.     |
|-----|-----------------|--------|---------|-------|-------|--------|
|     |                 |        |         |       | M.m.  |        |
| 1.  | At b.p. of O .. | -182.5 | 19.631  | 14.4  | 758.5 | 1.1181 |
| 2.  | At b.p. of N .. | -195.5 | 20.536  | 14.4  | 758.5 | 1.1700 |
| 3.  | At m.p. of N .. | -210.5 | 21.743  | 14.55 | 758.5 | 1.2386 |
| 4.  | At b.p. of H .. | -252.5 | 25.003  | 14.6  | 758.5 | 1.4256 |
|     |                 |        | (solid) |       |       |        |

Where T' = the temperature Centigrade of the condensed gas in the flask at the time of observation.

V = volume of gas in litres at temperature T° C. and pressure p m.m.

d = density of the condensed gas at T° C.

The volume of the flask at 15° C. was 23.9212 c.c.

Coefficient of expansion of glass, taken as 0.0000250.

Weight of 1 litre of oxygen at 0° C. and 760 m.m., taken as 1.430 grms.

TABLE II.—Nitrogen.

| No. | Description.    | T'.    | V.      | T.    | p.   | d.     |
|-----|-----------------|--------|---------|-------|------|--------|
|     |                 |        |         |       | M.m. |        |
| 1.  | At b.p. of N .. | -195.5 | 15.192  | 16.1  | 747  | 0.8042 |
| 2.  | At m.p. of N .. | -210.5 | 16.592  | 15.95 | 747  | 0.8792 |
| 3.  | At b.p. of N .. | -252.5 | 18.911  | 14.65 | 761  | 1.0265 |
|     |                 |        | (solid) |       |      |        |

The same notation is used as in Table I.

The volume of the flask at 14.2° C. was 22.1454 c.c.

Coefficient of expansion of glass, 0.0000250.

Weight of 1 litre of nitrogen at 0° C. and 760 m.m., 1.2564.

TABLE III.—Hydrogen.

| No. | Description.    | T'.     | V.      | T.   | p.   | d.      |
|-----|-----------------|---------|---------|------|------|---------|
|     |                 |         |         |      | M.m. |         |
| 1.  | At b.p. of H .. | -252.5  | 18.051  | 14.6 | 760  | 0.07004 |
| 2.  | H under 76 m.m. | -258.29 | 19.447  | 14.6 | 760  | 0.07544 |
| 3.  | H under 10 m.m. | -259.9  | 19.597  | 14.3 | 762  | 0.0763  |
|     |                 |         | (solid) |      |      |         |

The same notation is used as in Table I.

The volume of the flask at 14.2° C. was 22.1454 c.c.

Coefficient of expansion of glass, 0.0000250.

Weight of 1 litre of hydrogen at 0° C. and 760 m.m., taken as 0.0899.

2. It is of advantage to estimate the effects of an error, or of any reading that may have been taken roughly. Taking the notation already employed, and putting *w* for the weight of 1 litre of the gas at 0° C. and 760 m.m., then the weight of gas used is—

$$W = \frac{V p 273}{760 (273 + T)} w;$$

and if *V*<sub>0</sub> be the volume of the flask up to the mark at some ordinary temperature T<sub>0</sub>° C., and *V*' its volume at T' the temperature Centigrade of the observations, then—

$$V' = V_0 \frac{1 + \beta T'}{1 + \beta T_0},$$

where β is the cubical coefficient of expansion of the glass. Hence, the density of the substance at the time of observation is—

$$d = \frac{W}{V} = \frac{V p 273 w}{760 (273 + T)} \times \frac{1 + \beta T_0}{V_0 (1 + \beta T')},$$

or, with sufficient accuracy for our investigation,—

$$d = \frac{273 w}{760 V_0} \times \frac{V p (1 + \beta T_0 - T')}{273 + T} \quad (1).$$

An important error might be expected to arise from an inaccurate knowledge of T', the temperature of the cooled gas. An alteration of a degree in the value of T' would alter the value of *d* by—

$$- \frac{273 w}{760 V_0} \times \frac{V p}{273 + T} \beta,$$

or very approximately, a rise of a degree in the estimate of T' above its true value would diminish *d* by β *d*—in the present case by 1/40000 part.

\* A Paper read before the Royal Society, March 17, 1904.

Similarly an error of a degree in the estimate of the temperature  $T_0$ , at which the volume of the flask was measured, would have the like small effect, but in the opposite direction.

On the other hand, errors in the measurements of either  $V$  or  $V_0$  or of  $p$  or  $T$  would give directly proportional errors in the value of  $d$ . The values of  $T$  were easily read to one-tenth of a degree, so that an error in this quantity would have an effect less than  $1/3000$  on the value of  $d$ . Similarly the pressure was read to  $\frac{1}{4}$  m.m., leaving an error of less than  $1/1500$  on the density. The volume  $V$  showed a tendency to error in the earlier portion of each group of experiments, but this was eliminated as the experiments progressed and especially where the results were more important, namely, in the region of the solidified gases. The greatest care was taken to insure that the whole volume of the bulb was occupied by the solid. The gas was allowed to enter in successive portions, each of which was liquefied and solidified previous to any further admission, so as to insure the absence of any vacua due to contraction.

3. The results for oxygen seem low, the boiling-point density being 1.118, whereas former results gave 1.138. On plotting the densities as ordinates to the temperatures as abscissæ the observations lie very closely on a straight line which (by least squares), is—

$$d = 1.5154 - 0.004420t \quad \dots \quad (2),$$

$t$  being absolute temperature.

Such a line as (2) must in any case be only a chord of the curve of liquid densities, and the nearer two observations are to the absolute zero, the more nearly will the chord joining them partake of the nature of a tangent to this curve at the absolute zero. Now, at so low a temperature as  $20.5^\circ$  for oxygen we may consider its gaseous density to be practically negligible. Hence one point on Matthias's rectilinear diameter for oxygen will be, at  $20.5^\circ$ , a density equal to 0.7128, the half of that given in No. 4, Table I. In former papers I found the density of liquid oxygen at its boiling-point to be 1.138, and of gaseous oxygen at the same point to be 0.00440; half the sum of these is 0.5712, which gives another point on Matthias's diameter at  $90.5^\circ$ . Thus Matthias's diameter for oxygen is—

$$d = 0.7543 - 0.002023t \quad \dots \quad (3),$$

and taking the critical temperature as  $155^\circ$  absolute we get the critical density to be 0.4407, agreeing notably with the usually accepted value 0.44. The inference from this is that the density of solid oxygen at the boiling-point of hydrogen is 1.4256.

The results for nitrogen, taken at three temperatures, do not warrant the deduction of a linear relation between  $d$  and  $t$ , especially as on plotting the observations, the concavity of the liquid density curve, though slight, is quite apparent. However, there are two observations at temperatures so low that the corresponding gaseous densities may be neglected, thus enabling us to construct a Matthias diameter. At the boiling-point of hydrogen, the ordinate of the Matthias line is therefore very nearly  $\frac{1}{2}$  ( $1.0265$ ) =  $0.5133$ ; similarly at the melting-point of nitrogen the ordinate is  $\frac{1}{2}$  ( $0.8792$ ) =  $0.4396$ . Hence the Matthias diameter is—

$$d = 0.5492 - 0.00175t \quad \dots \quad (4),$$

which for the critical temperature  $127^\circ$  gives the critical density as 0.3269. This agrees very well with the value deduced by Matthias (*Mem. Soc. Roy. des Sci. de Liège*, 3rd Series, 1899) from Wroblewski's liquid densities, namely 0.333, though it is somewhat higher than the value 0.299 which he deduced from the theory of corresponding states ("Le Point Critique des Corps Purs," p. 176).

Only three observations have been obtained for hydrogen which again lie nearly on a straight line, but nevertheless present a very slight concavity to the axis of temperature. If we treat the two lowest densities as we have done with nitrogen, we get for the Matthias diameter, the line—

$$d = 0.04136 - 0.000247t \quad \dots \quad (5),$$

whence the annexed table of critical densities according to the—

| $t_c$      | $d_c$   |
|------------|---------|
| $28^\circ$ | 0.03444 |
| 29         | 0.03420 |
| 30         | 0.03395 |
| 31         | 0.03370 |
| 32         | 0.03346 |
| 33         | 0.03321 |
| 34         | 0.03296 |

temperatures chosen for the critical temperatures. Berthelot gives an estimate for the critical density as 0.033, and quotes Wroblewski's critical temperature as  $33^\circ$ , two results closely in accord with the numbers in this table. We are, therefore, justified in considering these hydrogen densities as very closely in agreement with facts, the density at the boiling-point coinciding nearly with my former determinations.

4. Assuming that vapour densities at very low temperatures may be neglected in comparison with corresponding liquid densities, the Matthias diameter enables us to approximate to the molecular volumes of the condensed gases at the absolute zero. For if  $d' = a - bt$  be the equation of Matthias's diameter, then  $d = 2a - bt$  is very approximately the tangent of the liquid density curve near the absolute zero, and therefore  $1/2a$  is the specific volume at absolute zero.

Hence from the above equations for Matthias's diameter, if  $V_0$  = the molecular volume at absolute zero, we have  $V_0 = 21.21$  for oxygen,  $V_0 = 25.49$  for nitrogen, and  $V_0 = 24.18$  for hydrogen. The oxygen and nitrogen molecular volumes at absolute zero probably err by defect; but the hydrogen result must be taken as very near the true value.

We may compare these values with the results of theoretical investigation. Guldberg gives for the molecular volume at zero of oxygen 21.5, and of nitrogen 23.6 (*Zeit. f. Phys. Chem.*, 1895, vol. xvi., p. 7); and Berthelot's values for the same gases respectively are 20.8 and 25.0 (*Comptes Rendus*, March, 1900). From Baly and Donnan's (*Journ. Chem. Soc.*, July, 1902, pp. 911–914) equations for oxygen, nitrogen, carbonic oxide, and argon, deduced from observations within the range of temperature  $69^\circ$ – $90^\circ$  absolute, we find the following values for the molecular volumes at absolute zero:—oxygen 20.30; nitrogen 24.04; carbonic oxide 24.54; argon 20.34.

Again, the Waterston-Avenarius formula connecting temperature and fluid volume, namely,—

$$v = c - d \log (A - t) \quad \dots \quad (6),$$

where  $A$  is the critical temperature, gives the following equations from Baly and Donnan's results,

$$\text{Oxygen} \dots v = 1.9305 - 0.5838 \log (154 - t),$$

$$V_0 = 20.90, \quad c/d = 3.3.$$

$$\text{Nitrogen} \dots v = 2.5659 - 0.7868 \log (127 - t),$$

$$V_0 = 25.80, \quad c/d = 3.27.$$

$$\text{Carbonic oxide} \dots v = 2.6181 - 0.7948 \log (133 - t),$$

$$V_0 = 26.04, \quad c/d = 3.29.$$

$$\text{Argon} \dots v = 1.6331 - 0.5026 \log (155 - t),$$

$$V_0 = 21.30, \quad c/d = 3.70.$$

This same formula has been adopted by Mallet and Friderich (*Arch. Sci. Phys., et Nat.*, July, 1902), subject to the modification that  $A$  is a unique temperature to be determined for each substance from experiment. On applying it to twenty-five substances, studied by Sydney Young, they find that  $A$  is somewhat higher than the critical temperature, and that  $c/d$  is always very nearly equal to 3.78. Baly and Donnan's observations give rise to these Waterston-Mallet equations:—

$$\text{Oxygen} \dots v = 3.88413 - 1.3604 \log (253 - t),$$

$$V_0 = 19.68, \quad c/d = 2.86.$$

$$\text{Nitrogen} \dots v = 3.1563 - 1.0560 \log (143.67 - t),$$

$$V_0 = 24.58, \quad c/d = 2.99.$$

$$\text{Carbonic oxide} \dots v = 4.60090 - 1.64207 \log (191.1 - t),$$

$$V_0 = 23.94, \quad c/d = 2.80.$$

$$\text{Argon} \dots v = 0.98285 - 0.19263 \log (112.4 - t),$$

$$V_0 = 23.52, \quad c/d = 5.10.$$

With assumed values for A, in the neighbourhood of the range of temperature in which we look for the critical temperature, two Waterston-Mallet formulæ were constructed for hydrogen based on the results of Table III., namely:—

$$v = 23.22 \cdot 7.536 \log(36.5 - t), V_0 = 22.90, \\ v = 26.83 \cdot 9.592 \log(41.5 - t), V_0 = 22.62.$$

Assuming that for liquids Van der Waals's equation may be written—

$$\frac{Rt}{v-b} = \frac{a}{v^2} \dots \dots (7),$$

an assumption which has been employed by G. N. Lewis (*Am. Acad. Arts and Sci.*, 1900, vol. xxxv., pp. 1-27) and others, the results of Table III. give for hydrogen, with this equation,  $b = 11.56$ , and hence  $V_0 = 23.12$ .

For comparison these values may be arranged in tabular form thus:—

|                            | O.    | N.    | H.    | CO.   | Arg.  |
|----------------------------|-------|-------|-------|-------|-------|
| Present experiments ..     | 21.21 | 25.49 | 24.18 | —     | —     |
| Guldberg .. .. .           | 21.50 | 23.6  | —     | —     | —     |
| Berthelot .. .. .          | 20.8  | 25.0  | —     | —     | —     |
| Baly, Linear .. ..         | 20.30 | 24.04 | —     | 24.54 | 20.34 |
| Baly, Waterston ..         | 20.90 | 25.80 | —     | 26.04 | 21.30 |
| Baly, Waterston-Mallet ..  | 19.68 | 24.58 | —     | 23.94 | 23.52 |
| Waterston-Mallet, for H .. | —     | —     | 22.90 | —     | —     |
| Waterston-Mallet, for H .. | —     | —     | 22.62 | —     | —     |
| Lewis .. .. .              | —     | —     | 23.12 | —     | —     |

The two Waterston-Mallet results for hydrogen are of importance as showing that even great variations in the value of A affect but little the zero molecular volume. It is further to be remarked that the hydrogen results claim precedence over the others because they have been obtained from observations extending down to some  $14^\circ$  absolute. Moreover, the value 24.18 for hydrogen has been got directly from experiment, whilst the other values have been obtained from semi-theoretical equations whose validity is subject to some doubt.

(To be continued).

# OBSERVATIONS ON SOME CONTINUOUS INTRAMOLECULAR, AND AT FIRST REVERSIBLE, CHANGES EXTENDING OVER PROLONGED PERIODS OF TIME.

By R. J. FRISWELL.  
(Concluded from p 197).

## PART II. *Experimental.*

On October 3rd, 1885, there were introduced into a small flask 0.25 grm. aminoazobenzene base in crystals, 5.0 grms. aniline hydrochloride crystals, and 15 c.c. water.

An almost colourless solution of the aniline salt was formed, in which the bright yellow crystals of aminoazobenzene base lay in a heap at the bottom. The flask was now rapidly raised to the boil for about one minute, and closely corked up.

The effect of the boiling was to cause the aminoazobenzene base to dissolve, forming the characteristic deep red solution exhibited by its hydrochloride. As the solution cooled, it became very much paler, and much aminoazobenzene base crystallised out as a bulky mass, while on the bottom of the flask were perhaps a dozen minute steel-blue needles of the hydrochloride.

The flask was put aside in a cool, badly lighted place, and when examined two days later, on the 5th, the steel-blue hydrochloride crystals had greatly increased in number. Daily examinations were made, and it was found that this change continued.

On the 12th, the base crystals were scarcely visible, and

on the 19th they had entirely disappeared, save some that had stuck to the sides of the flask above the liquid, and the whole lower part was full of the hydrochloride.

On the same day (October 3rd), another similar flask, No. 2, was started with the same quantities of the materials, but it was at once put aside in the cold without heating.

The same change occurred, though more slowly, but by the 19th the whole of the base crystals had vanished, and were replaced by exceedingly minute crystals of the hydrochloride.

On October 5th, another flask, No. 3, was started, an exact repetition of No. 2. It behaved in exactly the same way.

Some weeks later, when all traces of the base had gone, this flask, No. 3, was raised to the boil. On cooling, much base was seen to be regenerated, and nine days later the crystals of base were still visible, though the conversion into hydrochloride was slowly taking place.

No. 4 flask, started on October 5th, contained 0.25 grm. aminoazobenzene base, 5 grms. aniline nitrate, and 15 c.c. water. On boiling it, no change occurred; both base and aniline nitrate crystallised out unchanged. They remained unchanged for many years.

No. 5 flask, started the same day, contained 0.25 grm. aminoazobenzene base, 5 grms. aniline hydrochloride, 0.25 grm. free aniline, and 15 c.c. water. It was boiled; the base crystallised out somewhat darker in colour.

No. 6 flask, on the same day, consisted of 0.25 grm. aminoazo-base, 5 grms. chloride of ammonium, and 15 c.c. water. On cooling, the base crystallised out in splendid needles, and it remained unchanged for nearly twelve years, when the flask was destroyed.

On October 10th, No. 7 flask was started with the aminoazo-base and free aniline in molecular proportions as well as aniline hydrochloride as follows:—Aminoazobenzene base, 0.5 grm; aniline, 0.24 grm.; aniline hydrochloride, 10 grms.; water, 30 c.c. It was boiled, and, on cooling, the base crystallised out rather darker than before. No change took place. No crystals of the hydrochloride were visible.

Accordingly, on December 3rd, after an interval of forty-two days, the contents of the flask were vigorously shaken, and then, as nearly as possible, one-half was poured into flask 8. To it was added a single clean crystal of the aminoazobenzene hydrochloride. Thirteen days later no change had taken place in either. They were therefore each raised to the boil for one minute, and again put aside.

On April 27th, 1886, 131 days later, they were still unchanged, not a crystal of aminoazobenzene hydrochloride could be seen.

They were put aside in a dark, cool place till March 2nd, 1887, when, on bringing them to the light, each was found with the lower part filled with large steel-blue lustrous crystals of aminoazobenzene hydrochloride.

On February 1st, 1886, flask No. 1, which had in nineteen days completely changed its base into chloride, had remained from its starting 120 days; it was raised to the boil, and on cooling it had returned to its original appearance after charging, boiling, and cooling, i.e., a little steel-blue hydrochloride lay on the bottom, above this a bulky mass of fine yellow crystals of the base.

These latter continued to diminish steadily, and seven days later, on the 8th, the base had entirely vanished, and the hydrochloride correspondingly increased. Thus the flask had twice gone through this series of changes.

The work which it has taken the amino-base fourteen days to do at the average temperature is undone by one minute's boiling. After that it takes six days at ordinary temperatures to re-enact the change.

The phenomena are most remarkable, when it is remembered that the effect of the boiling is to hasten the first stages of the change, for in flask No. 1 crystals appeared as soon as it was cool, while in flask 3, which was not boiled, several days elapsed before any showed themselves.

TABULAR VIEW OF EXPERIMENTS.

| Substances engaged in reaction.                                                                                    | Date of commencement<br>Into amidoazobenzene hydro-<br>chloride. | Date of completion                           | Number of days.                                                                                                                         | Character of vessel. | Remarks.                                                |
|--------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------|----------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------|----------------------|---------------------------------------------------------|
| 1. $C_{12}H_{11}N_3$ , 0.25 grm.<br>$C_6H_5NH_2HCl$ , 5.0 grms.<br>$H_2O$ , 15 c.c.                                | Oct. 3, 1885.                                                    | Oct. 19, 1885.                               | 16.                                                                                                                                     | Corked flask.        | Boiled for a few seconds.                               |
| 2. Same as No. 1.                                                                                                  | Oct. 3, 1885.                                                    | Oct. 19, 1885.                               | 16                                                                                                                                      | Corked flask.        | Entirely in the cold.                                   |
| 3. Same as Nos. 1 and 2.                                                                                           | Oct. 3, 1885.                                                    | Oct. 19, 1885.                               | 16                                                                                                                                      | Corked flask.        | As No. 1 ( <i>i.e.</i> , boiled).                       |
| 4. $C_{12}H_{11}N_3$ , 0.25 grm.<br>$C_6H_5NH_2HNO_3$ , 5.0 grms.<br>$H_2O$ , 15 c.c.                              | Oct. 3, 1885.                                                    | Unknown.                                     | Over 550.                                                                                                                               | Corked flask.        | In about 3000 days colour had formed.                   |
| 5. $C_{12}H_{11}N_3$ , 0.25 grm.<br>$C_6H_5NH_2HCl$ , 5.0 grms.<br>$C_6H_5NH_2$ , 0.28 grm.<br>$H_2O$ , 15 c.c.    | Oct. 3, 1885.                                                    | Unknown.                                     | Unknown.                                                                                                                                | Corked flask.        | Experiment lost.                                        |
| 6. $C_{12}H_{11}N_3$ , 0.25 grm.<br>$NH_4Cl$ , 5.0 grms.<br>$H_2O$ , 15 c.c.                                       | Oct. 5, 1885.                                                    | No change.                                   | No change after 4280 days.                                                                                                              | Corked flask.        | Boiling had no effect.                                  |
| 7—8. $C_{12}H_{11}N_3$ , 0.5 grm.<br>$C_6H_5NH_2HCl$ , 10.0 grms.<br>$H_2O$ , 30 c.c.<br>$C_6H_5NH_2$ , 0.24 grm.  | Oct. 10, 1885.                                                   | March 2, 1887.                               | All crystals in 405 days.                                                                                                               | Corked flask.        | Divided into two.                                       |
| X. $C_{12}H_{11}N_3$ , 0.5 grm.<br>$2(C_6H_5NH_2)H_2SO_4$ , 2.0 grms.<br>$H_2O$ , 15 c.c.                          | Mar. 3, 1887.                                                    | No change.                                   | No change after 3900 days. None after 17 years.                                                                                         | Corked flask.        | Boiling no effect.                                      |
| Y. $C_{12}H_{11}N_3$ , 1.0 grm.<br>$C_6H_5NH_2HCl$ , 20 grms.<br>$H_2O$ , 60 c.c.                                  | Mar. 2, 1887.                                                    | Unknown.                                     | Very slow.                                                                                                                              | Corked flask.        | Cold. Still—after 3600 days—many fine crystals of base. |
| Tube 1. $C_{12}H_{11}N_3$ , 0.5 grm.<br>$C_6H_5NH_2HCl$ , 5 grms.<br>$H_2O$ , 15 c.c.                              | Mar. 12, 1887.                                                   | Jan. 30, 1888.<br>$t$ 15—17°.                | 325 days all chloride.                                                                                                                  | Sealed tube.         | Boiled.                                                 |
| Tube 2. $C_{12}H_{11}N_3$ , 0.5 grm.<br>$C_6H_5NH_2HCl$ , 10 grms.<br>$C_6H_5NH_2$ , 24 drops.<br>$H_2O$ , 15 c.c. | Mar. 15, 1887.                                                   | Kept in well till Jan. 30, 1888. $t$ 10—11°. | 320 days, no change.                                                                                                                    | Sealed tube.         | Boiled.                                                 |
| Tube 3. $C_{12}H_{11}N_3$ , 0.5 grm.<br>$C_6H_5NH_2HCl$ , 10 grms.<br>$H_2O$ , 15 c.c.<br>$C_6H_5NH_2$ , 24 drops. | Mar. 14, 1887.<br>Changed into colour.<br>Boiled and sealed.     | Unknown.                                     | Colour formed.                                                                                                                          | Sealed tube.         | Kept at about 49° C.                                    |
| Tube 4. $C_{12}H_{11}N_3$ , 0.25 grm.<br>$C_6H_5NH_2HCl$ , 5 grms.<br>$H_2O$ , 15 c.c.                             | May 16, 1894.<br>Not boiled, but sealed cold.                    | August, 1895.<br>All hydrochloride—440 days. | Colour is not decided in Oct., 1897—say, in 1219 days,—but June 4, 1900, gone almost as far as No. 4; Nov., 1900, quite gone—2370 days. |                      | Colour suspected in middle of September, 1897.          |
| $C_{12}H_{11}N_3$ , 0.25 grm.<br>$C_6H_5NH_2HCl$ , 5 grms.<br>$H_2O$ , 20 c.c.                                     | May 16, 1894.<br>Boiled, and sealed hot.                         | Jan. 1, 1896.<br>Nearly all hydrochloride.   | Colour suspected in June, 1897 ( <i>i.e.</i> , in, say, 770 days); undoubted in Sept., 1897; and complete March, 1898—say, 1219 days.   |                      |                                                         |

We have here a change which takes place against what must be an ever increasing resistance generated by the process of its own increase; for just as an increasing weight pressing on a confined volume of gas meets with a resistance continually growing, so in this case the amidoazo base continues to rob the aniline chloride of its acid, thereby setting free aniline in continually increasing quantity, yet aniline can, when present in sufficient amount, totally prevent the change.

That there is, however, a limit at which, though it can enormously retard, it cannot stop the change, is shown by the remarkable history of flasks 7 and 8. In the first of these for forty-two days there is no change. It is then divided, and by being boiled is given a fresh start; for 131 days there is no change, and yet after a further interval of 308 days the change is complete. This flask contained a molecule of free aniline in addition to the molecule that would be set free by the complete conversion of the aminoazo base into hydrochloride. Hence it follows that a molecule of free aniline cannot prevent a molecule of aminoazobenzene base from robbing a molecule of aniline hydrochloride of its acid, though it can prolong the time required for this change from somewhat less than sixteen days to a time longer than 131 days, but less than 439 days. And from Experiment 5, that about two molecules of free aniline can either altogether stop the change, or prolong it for a period at least longer than 510 days.

It is to be observed that in Experiments 7 and 8 at the termination of the change there are in solution two molecules of free aniline.

The variation introduced in No. 8, viz., the introduction of a crystal of the hydrochloride, appears to have no effect.

### PART III.

When the experimental work had gone so far, viz., in 1887, various causes combined to lead to its being put aside. It had been suggested that the changes might be due to the gradual evaporation of aniline through the corks with which the flasks were closed. Accordingly, a series of experiments was set going in sealed tubes. These comprised the foregoing series, except with the nitrate and sulphate, which had shown no sign of change.\* This second series behaved as the first had done, and thus settled the question against the change being due to volatility of the base.

The experimental flasks and tubes was still kept, and observed from time to time, as another change was coming on, though so slowly, that it had during an interval of inattention crept on so far that the approximate time of its inception was lost. This was the formation of colour, which began both in sealed tubes and in flasks. Accordingly, in May, 1894, I again prepared some tubes, and on the 16th prepared two. No. 4 contained 0.25 of aminoazobenzene base, 0.5 grm. aniline hydrochloride, and 15 c.c. water. Great care had, as before, been taken in preparing the aminoazobenzene base, and the aniline salt was twice re-crystallised from water.

The sealed tube was placed in a cardboard box on a shelf in the laboratory, where its temperature probably did not vary greatly from 15° C. The change went on in its usual course, and in about 450 days, i.e., in August, 1895, it was judged to be completely converted into hydrochloride.

In September, 1897, or say 1200 days, there were probable indications of the beginning of the colour formation stage. On the same day the second tube, containing 0.25 grm. aminoazobenzene and 20 c.c. water, was also prepared. This tube was raised to the boiling-point for an instant, and sealed. The usual phenomena, viz., the red solution of the aminoazobenzene hydrochloride, while hot, the reformation and crystallisation of the aminoazobenzene base on cooling, were observed.

In August, 1895 (450 days), when the change in the

other tube was judged to be complete, this exhibited only a partial formation of the hydrochloride, and the total disappearance of the base was not considered to have taken place, even in January, 1896, or in 600 days, which might perhaps be considered as due to the greater dilution of the solution.

But since then the colour change began to develop. I judged this to begin in the early part of August, 1896, or in about 710 days. It then appeared to go with greater rapidity. In September, the reddish stage had been reached, and at the end of that month it had advanced so rapidly that no crystals of hydrochloride could be seen, and bronze shells of colour were showing.

The bluish stage was soon reached, and in March, 1898, appeared to be complete, and I judged that all was changed into colour by November, 1900.

It will therefore be seen that this change is one which at ordinary temperatures requires more than four, but less than ten, years for its accomplishment.

Though the hydrochloride of aniline gives up its acid to the base in a period varying from nineteen to 460 days, according to the absence or presence of free aniline at the initial stage of the change, chloride of ammonium does not do so, even in a period of 4000 days, and, moreover, even when boiled with the base, shows no signs of parting with acid to it.

Lastly, comes the long delayed change into colour, a colour belonging to the indulin group.

After many years I have at last, in the tube No. 4, caught the commencement of the change, and shown that the time for reaction at an average temperature of 15° is to the time at 100° as somewhere about 4900 to 1, as far as the colour is concerned.\*

But for the conversion into the hydrochloride, the time is as the time taken for boiling, say five minutes to 440 days or 1:126520. By raising to the boil, this work is undone; for on cooling, base separates, again to go back into the hydrochloride, and this series of changes would appear to be perpetually possible but for the appearance of the colour reaction.

### NOTE TO TABLE.

The effect of the boiling is first to convert into hydrochloride of aminoazobenzene, which decomposes on cooling, the base separating, but if the boiling is continued for six hours, colour is formed, and all, or nearly all, aminoazobenzene is gone. Nevertheless, an unboiled tube goes quicker into hydrochloride crystals, and slower into colour than a boiled tube, and a tube once converted into hydrochloride crystals is restored partly to base by boiling and cooling. For colour formation (after momentary boiling) the time relation is six hours boil, = 29,256 hours at 15–25° or 1:4866.

All the tubes and flasks, except those with ammonium chloride, aniline sulphate, and aniline nitrate, were showing colour at some period before 1893.

Royal Institution.—On Tuesday next, May 3, at 5 o'clock, Mr. L. Fletcher will deliver the first of three lectures at the Royal Institution on "Meteorites"; on Thursday, May 5, at the same hour, Mr. Arthur Hassall commences a course of three lectures on "Great Britain and Europe (1763-1793)"; and on Saturday, May 7, at 3 o'clock, Mr. Donald F. Tovey begins a course of three lectures on "Sonata Style and the Sonata Forms," with Musical Illustrations. The Friday Evening Discourse on May 6 will be delivered by Dr. Chalmers Mitchell on "Anthropoid Apes"; on May 13 by Mr. M. H. Spielmann on the "Queen Victoria Memorial"; and on May 20 by Prof. Rutherford on the "Radiation and Emanation of Radium."

\* This colour was investigated by Istel (*Chem. Zeit.*, xiv., 1535). He considered that complete conversion was attained in twenty-four hours at 70–80°. In addition to the soluble colour, he obtained an insoluble one resembling the nigrosines, and identified azophenine as existing among the products of the reaction.

\* The latter was kept for about seventeen years, but never changed, and it was therefore destroyed.

## THE ELEMENTS: VERIFIED AND UNVERIFIED.

By CHARLES BASKERVILLE, Ph.D., F.C.S., Smith Professor of General Chemistry, University of North Carolina.

(Concluded from p. 195).

## LIST OF PROPOSED CHEMICAL ELEMENTS (continued).

| Date. | Name.               | Source.                                 | Authority.                | Reference                                                                                                                           | Remarks.                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
|-------|---------------------|-----------------------------------------|---------------------------|-------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1901  | Euxenium earth I.   | Euxenite from Bre-<br>vig.              | Hofmann and Prandtl.      | Berichte, 34, 1064.                                                                                                                 | Differs from zirconium in certain reactions. Atomic weight 178 (circa) if it be tetravalent. Also another unknown substance present, giving chemical reactions quite different from I. Giesow and Horkheimer failed to secure either from purchased pure zirconium sulphate (Zeit. Anorg. Chem., 1902, 32, 372).                                                                                                                                                                     |
| 1901  | Euxenium earth II.  | Euxenite from Bre-<br>vig.              | Hofmann and Prandtl.      | Berichte, 34, 1064.                                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| 1902  | <i>Amarillium</i> . | Copper ore (British<br>Columbia).       | Courtis.                  | Trans. Am. Inst. Min. Eng.,<br>advance sheet, New Haven<br>Meeting, October, 1903.                                                  | In parting gold button observed peculiar behaviour. Hille-<br>brand says ("Review Am. Ch. Res., 1901, 9, 151) :—<br>"Eastern assayers had reported the metal in the ore<br>to be platinum and palladium" (?).                                                                                                                                                                                                                                                                        |
| 1903  | <i>Britium</i> .    | Coal ashes.                             | Reporter's fertile brain. | Washington Post, Nov. 18.                                                                                                           | Special correspondence from Newark, N. J. Reported<br>presence of material in coal ashes which produces more<br>heat when placed under furnace than fuel bought from<br>trust.                                                                                                                                                                                                                                                                                                       |
| 1903  | "Radium-foil."      | Zinc-bearing<br>minerals.               | Kunz and Baskerville.     | Am. Journ. Sci., 1904 (see<br>next).                                                                                                | Zinc oxide and sulphide, willemite, and kunzite (which<br>contains zinc) fluoresce and phosphoresce with Röntgen<br>rays, ultra-violet light, radium, and actinium prepara-<br>tions, even shielded.                                                                                                                                                                                                                                                                                 |
| 1903  | New element (?).    | Minerals from Mono<br>Lake, California. | Kunz and Baskerville.     | New York Acad. Sci., Octo-<br>ber 6; Am. Journ. Science,<br>January, 1904; Science,<br>18 (N.S.), 769.                              | Every mineral, without exception and regard to chemical<br>composition, from this lake phosphoresces with ultra-<br>violet light. Considering the formation of these minerals,<br>they appear to have some common constituent giving<br>this response. Whether it be new cannot be stated,<br>but the observations are sufficiently unique to place on<br>record for subsequent investigation. See the full paper<br>for other somewhat similar observations with other<br>minerals. |
| 1903  | Tellurium X.        | Tellurium.                              | Pellini.                  | Gazz. Chim. Ital., 33, 11, 35.                                                                                                      | Maintains presence of constant small radio-active con-<br>stituent with atomic weight about 212, which causes<br>tellurium to hold its present anomalous position in the<br>Periodic Table. (See Marckwald, Ber., 35, 2285).                                                                                                                                                                                                                                                         |
| 1903  | Berzelium.          | Thorium.                                | Baskerville.              | Presented before N. Carolina<br>Section, American Chemi-<br>cal Society, November 28;<br>American Assoc. Adv. Sci.,<br>December 29. | Obtained by fractioning thorium chlorides during production<br>by volatilisation. "Weisser dampf" of Berzelius, hence<br>name. Oxide does not phosphoresce with ultra-violet<br>light, while thorium oxide does. Atomic weight (im-<br>pure material) 212.7, when regarded tetravalent.                                                                                                                                                                                              |



LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MARCH 31ST, 1904.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, ESQ., M.Inst.C.E.,  
Water Examiner, Metropolis Water Act, 1871.

London, April 11th, 1904.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 219 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from March 1st to March 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 219 samples examined by us during the month, all were clear, bright, and well filtered.

There has been a deficit in the rainfall at Oxford during March; the actual amount of rain measured during the past month was 1.95 inches, and the average fall is 1.47 inches; thus we have a deficit of 0.42 inch, which, if subtracted from the previous excess of 2.20 inches, leaves an excess of 1.78 inches, or 33.3 per cent above the thirty-five years' average.

Our bacteriological examinations of 401 samples taken during the month have given the results recorded in the following table. Besides these samples we have examined 387 others from special wells, standpipes, &c., making 788 samples in all:—

|                                                                                                                 | Microbes<br>per c.c. |
|-----------------------------------------------------------------------------------------------------------------|----------------------|
| New River, unfiltered (mean of 27 samples) ..                                                                   | 210                  |
| New River, filtered (mean of 80 samples) ..                                                                     | 11                   |
| Thames, unfiltered (mean of 27 samples) ..                                                                      | 7410                 |
| Thames-derived water from the clear-water<br>wells of eight Thames-derived supplies (mean<br>of 213 samples) .. | 17                   |
| Ditto ditto .. .. . highest                                                                                     | 310                  |
| Ditto ditto .. .. . lowest                                                                                      | 0                    |
| River Lea, unfiltered (mean of 27 samples) ..                                                                   | 187                  |
| River Lea, from the East London Water Com-<br>pany's clear-water wells (mean of 27 samples)                     | 9                    |

Of the 320 daily samples taken from the general filter wells of the Metropolitan Water Companies, nineteen samples, or 5.9 per cent, were sterile. Ten samples, or 3.1 per cent, contained more than 100 microbes, and of these, only three samples contained more than 150 microbes per c.c. The ten excess samples contained an average of 148 microbes per c.c. In February twenty-seven excess samples contained an average of 162 microbes per c.c.

Both the Thames and the Lea have now recovered from the effects of the abnormal floods which have been prevalent for so many months. The result is that both the chemical and bacteriological condition of the water supply has been manifestly improved, and may now be characterised as excellent for this period of the year.

We are, Sir,

Your obedient Servants,  
WILLIAM CROOKES.  
JAMES DEWAR.

HAUSMANNITES FROM SWEDEN.

By A. GORGEU.

In a previous paper on "The Native Oxides of Manganese" (*Bull. Soc. Chim.*, 1893, p. 650), I found from 1 to 8 per cent of oxide of zinc in the Hausmannites from Ilmenau in Thuringia, and I observed that this oxide replaces an equivalent amount of manganous oxide in the saline oxide,  $Mn_2O_3, MnO$ .

Since that time I have been able to procure, by the help of M. G. Flint, a mineralogist at Stockholm, several beautiful samples of the same mineral coming, one from Jacobsberg, and the two others from Langbanshyttan.

With these specimens I determined to try and find out whether I could still detect the protoxide of zinc or another oxide, playing the same part in the constitution of the mineral as oxide of zinc does in that from Ilmenau.

In the samples analysed, the gangue, consisting of crystals of dolomite and Richterite, was easily eliminated.

A single fine crystal, weighing 10 grms., furnished the sample for the analysis of Hausmannite from Langbanshyttan, No. 1; that of No. 2 from Jacobsberg was done on several very good isolated crystals; that of No. 3 from Langbanshyttan, consisted partially of crystals and partially of the massive portion which held them, without any gangue being apparent.

In the following table, opposite the figures given by the analysis, I have given the amount of oxygen contained in each of the acid or basic elements that the figures represent.

|                                   | No. 1.<br>Langbanshyttan. |         | No. 2.<br>Jacobsberg. |         | No. 3.<br>Langbanshyttan. |         |
|-----------------------------------|---------------------------|---------|-----------------------|---------|---------------------------|---------|
|                                   | Analysis.                 | Oxygen. | Analysis.             | Oxygen. | Analysis.                 | Oxygen. |
| H <sub>2</sub> O ..               | —                         | —       | 0.04                  | —       | —                         | —       |
| CO <sub>2</sub> ..                | 0.37                      | 0.27    | 0.50                  | 0.36    | 0.19                      | 0.14    |
| Richterite.                       | 0.20                      | —       | 0.23                  | —       | 0.90                      | —       |
| O ..                              | 6.52                      | 6.52    | 6.34                  | 6.34    | 6.54                      | 6.54    |
| MnO ..                            | 86.52                     | 19.50   | 85.79                 | 19.33   | 87.34                     | 19.68   |
| Fe <sub>2</sub> O <sub>3</sub> .. | 4.30                      | 1.29    | 5.75                  | 1.92    | 3.47                      | 1.04    |
| ZnO ..                            | —                         | —       | 0.22                  | 0.04    | 0.70                      | 0.14    |
| PbO ..                            | 0.55                      | 0.06    | 0.12                  | 0.01    | —                         | —       |
| BaO ..                            | —                         | —       | —                     | —       | 0.07                      | 0.01    |
| CaO ..                            | 0.43                      | 0.12    | 0.48                  | 0.14    | 0.44                      | 0.13    |
| MgO ..                            | 1.00                      | 0.40    | 0.44                  | 0.17    | 0.30                      | 0.12    |
| Alkalis ..                        | —                         | —       | 0.16                  | 0.03    | 0.45                      | 0.09    |
|                                   | 99.89                     |         | 100.07                |         | 100.40                    |         |

Considering only the peroxide of manganese that these three samples of Hausmannite contain, we notice that the ratios which exist between the weight of the oxygen of the protoxide of manganese, and that of the oxygen in those oxides above the protoxide, are as follows:—

3 : 0.991 in No. 1  
3 : 1.04 in No. 2  
3 : 1.01 in No. 3

—ratios which are all very close to 3 : 1, which theory requires for pure Hausmannite.

But, associated with this peroxide, we find 5 to 7 per cent of different metallic oxides which ought not to be neglected, and of which it may be of use to determine the rôle. Should we look upon them as impurities deposited at the moment of crystallisation of the Hausmannite? This supposition would be very acceptable if the samples taken were from massive or pulverulent specimens; we cannot, however, admit it easily for samples 1 and 2, the mean results of which are obtained from a single crystal or form a small number of very good crystals. Another hypothesis is possible, and seems to be more natural; it consists of assuming that, in the Swedish Hausmannites, the sesquioxide of manganese and the sesquioxide of iron form the acid portion of the mineral, and that its basic portion comprises more particularly the protoxide of manganese, and to a small extent the other protoxides revealed by analysis.

To justify this second supposition we ought to find the

same ratio, 3:1, between the quantities of oxygen in the two peroxides and the total weight of oxygen in the protoxides, as in the pure Hausmannite.

The necessary calculations have been made for the three minerals analysed, using the figures given in the second columns of the analyses.

We determined, for each sample, the weight of the sesquioxide of manganese it contained, taking the figure for the oxygen above the protoxides; the oxygen contained in the actual amount of sesquioxide found added to that of the oxygen contained in the ferric oxide furnished the total amount of oxygen in the acid portion of each mineral.

The weight of the sesquioxide of manganese being known, it was easy, with the help of the figures found by the analysis, to estimate that of the protoxide of manganese combined with it; to the weight of the oxygen contained in this latter we added that contained in the other protoxides. The total of these figures should be diminished by the oxygen required to saturate the carbonic acid, so as to get exactly the amount of oxygen in the basic portion.

By proceeding in this manner we found the following ratios, in the three Hausmannites, between the atoms of oxygen in the acid elements and those in their basic elements:—In the Hausmannite from Langbanshyttan, No. 1, the ratio was 3:0.994; in that from Jacobsberg, No. 2, the ratio was 3:0.99; and in that from Langbanshyttan, No. 3, the ratio was 3:1.02. These ratios are all as close to the theoretical figures, 3:1, as those found in the peroxides of manganese considered by themselves.

This manner of regarding them is confirmed by the synthetical experiments described in a paper which will appear shortly in the *Bulletin*.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 23.

## PROCEEDINGS OF SOCIETIES.

### PHYSICAL SOCIETY.

Ordinary Meeting, April 22nd, 1904.

Dr. R. T. GLAZEBROOK, F.R.S., President, in the Chair.

A PAPER entitled "Calculation of Colours for Colour Sensitometers and the Illumination of 'Three Colour' Photographic Transparencies by Spectrum Colours" was read by Sir W. DE W. ABNEY.

In three-colour photography, photographs have to be taken through a red, a green, and a blue screen, the transparencies or prints from which are then viewed. The exact shades and hues of these screens depend on the light which is used for viewing the transparencies or on the colours employed in printing. The present paper confines itself to the former case. It is well to settle the colours which should be used by a reference to the spectrum, remembering that if any other colours, such as those transmitted through glasses, are employed, they must inevitably be less pure. The true guide to selection must be founded on the trichromatic theory of colour-vision, and the author has shown in a former paper that the position of the colours in the spectrum can be fixed with extreme accuracy. Any part of the spectrum below C towards A gives a colour which excites no other sensation than the red sensation. The green and the blue sensations are felt unmixed with any other colour-sensation, though rendered impure by the sensation of white, at *b* for the former, and near the blue lithium line in the spectrum of lithium for the latter. Any departure from these positions causes an excess of colour due to one of the three sensations, and is not therefore advisable to adopt it, as it increases the impurity of the mixed colours by adding still more and quite unnecessary white to the resulting hue. These three colours form the theoretical starting-point for the determination of the exact hue of the screens which have to be used. The determination of the exact hues of the screens is one which

presents some difficulties if resort is confined to the spectrum. A perfect photographic plate is one on which, if a spectrum be thrown, the opacities of deposit will represent luminosity exactly, or the transparency taken from such a negative will transmit white light at every portion of the image corresponding to the luminosity of the spectrum at that point. A perfect photographic plate does not exist, and hence we are driven to a plan which, not absolutely exact, is yet as near exactitude as possible. Any intermediate colour between the three standard colours can be matched in hue and luminosity by mixing the red and green together in proper proportions, or the green and blue. The colours of pigment can be matched by a mixture of two of the three chosen colours. The author has shown how, by means of luminosity equations, it is possible to arrange two pigments so that the components of any standard colour, say the green, are equal. Photographing the two pigments in which the green components are made equal, we must find some medium which will allow only such rays to pass as will make the two images of equal opacity. This will be a green of some kind. With more colours similarly treated we get an approach to colours at different parts of the spectrum, and we can then determine the screen to be used for any kind of plate which it is possible to use. The amounts of red, green, and blue existing in the red, yellow, green, and blue pigments and white were determined, and the luminosities being known the amounts of each of three spectrum-colours in luminosities were determined to make up the total luminosity of the pigment (or white). Supposing that a screen was sought for which would give the proportions of red on the photographic plate. The luminosities had to be so reduced in the pigments chosen that the red component in each was of the same luminosity. This was effected by making rings of the colours on a disc and cutting off part of the rings by black. We thus have a disc in which all the rings have equal red luminosity. Having got three separate discs in which the red, the green, and the blue components are made equal respectively, the screens for the camera were found by altering the hue till the opacities in the negative of all the rings were equal. Having obtained the negatives, transparencies are made from them. These are usually placed in a triple lantern with the three colours behind the objective. The light coming through each is so arranged that the white formed by their mixture is of the same quality as sunlight; but as they are impure colours they give more white in the picture than is to be found in nature. Sir William Abney showed how to illuminate the transparencies by using spectrum-colours, placing slits on the three colours which answer best to the three colour-sensations.

A paper on "Normal Piling as connected with Osborne Reynolds's Theory of the Universe," was read by Prof. J. D. EVERETT.

The closest arrangement of equal spheres in *plano* is the triangular one in which each is touched by six. To obtain the closest packing in space, the pile of spheres must consist of a succession of these tiers.

In building such a pile, the laying of each successive tier gives a choice between two different positions of this tier relative to its predecessor. Suppose two regular tetrahedrons to be placed base to base, so that one is the reflection of the other relative to the base; and let the edges of one of these tetrahedrons be parallel to the six lines which join the centres of four spheres in mutual contact (say in the first and second tiers). The peculiarity of "normal piling" is that this rule holds in passing from each tier to the next, through the whole pile. The other alternative is represented by the edges of the second tetrahedron, and gives equally close packing. If we use the two tetrahedrons alternately as our guides in laying successive tiers, we obtain the system of closest piling which comes next in simplicity to normal piling. It may be called "anti-normal piling."

In normal piling every sphere belongs to six lines of spheres which are continued through the whole pile. They

are parallel to the six edges of the standard tetrahedron. In anti-normal piling there are only three such lines instead of six.

In normal piling every sphere belongs to four sets of triangularly arranged tiers, which are parallel to the four faces of the tetrahedron. In anti-normal piling there is only one set, instead of four.

Again, in normal piling there are three sets of squarely arranged tiers, their planes being at right angles. In anti-normal piling there are only fragmentary traces of square arrangement.

Prof. O. Reynolds's theory asserts that the universe is composed of infinitely hard equal spherical grains in approximately normal piling, which, on striking, rebound without loss of relative velocity; their free paths being infinitesimal compared with their diameters, except in places where the grains are out of gear (called surfaces of misfit). A spherical surface of misfit is the simplest kind of atom of ordinary matter, and is propagated as a solitary wave through the surrounding grains, by the transfer of grains across it in the reverse direction.

The excitation of electricity by friction is explained as the tearing away of a cluster of grains from one part of a normal pile and thrusting it into another part. Disarrangements of opposite kinds are produced at the two places, each involving diminished closeness of packing. Magnetism is explained as a rotational displacement of a spherical cluster of grains, sufficient to cause grains near the equator of the sphere to slip a tooth (as it were) in their gearing with the grains outside, while grains nearer the poles have not moved far enough to slip a tooth. The spherical cluster is thus subjected to rotational strain and stress.

Attractions and repulsions are manifestations of a tendency of disarranged grains towards closer packing; a tendency due to the continual impacts from without, which (as in the kinetic theory of gases) are equivalent to external hydrostatic pressure.

The paper maintains that, in a struggle for existence between different kinds of closest piling, represented by separate clusters with room to change their arrangements, normal piling possesses great advantages, first, in its six sets of lines of spheres, which serve as battering rams, and secondly, in its four sets of tiers in closest array, which facilitate the coalescence of adjacent clusters.

A "Note on the Diffraction Theory of the Microscope as applied to the case when the object is in motion," by Dr. R. T. GLAZEBROOK, was taken as read.

According to the Abbe theory of microscopic vision, when a grating is placed on the stage of a microscope and illuminated by plane waves, diffraction images are formed in the focal plane of the object-glass and the images in the view-plane result from these, and this is undoubtedly true. The following difficulty has, however, been raised: if the grating be moved in its own plane in a direction perpendicular to the ruling, the diffraction images do not change, those seen in the view-plane move; how then can the latter images be due to the former? The answer lies in the fact that in the above argument the effect of the differences of phase among the diffracted images has been neglected. The diffracted images are not all in the same phase, their relative phases are altered by shifting the grating, and the image pattern in the view-field is altered in consequence. A simple case is considered in the paper, and it is proved that the image in the view-plane may change without an alteration in the position of the diffracted images.

An Automatic Gas-pump was exhibited by Mr. C. E. S. PHILLIPS.

The apparatus is constructed upon a plan which enables the pump, when once set in operation, to continue automatically and to produce as perfect a Torricellian vacuum as is possible. It has been devised with a view to providing a comparatively portable machine suitable for special laboratory work, or for researches requiring pro-

longed pumping, and consists of three distinct parts, viz.: a small motor-driven mechanical pump, a four-way control valve, and a modified Toepler apparatus by which the final vacuum is obtained. Experiment has shown that the apparatus is fairly rapid in its action. In a preliminary trial a Röntgen-ray bulb of 200 c.c. capacity was exhausted in half an hour. The glass work is easily removed for cleaning or repairs, and the wood supports are fitted with adjustable brackets. The height of the complete apparatus is 18 inches.

There was also an exhibition of spectroscopic and other scientific apparatus by Mr. PETER HEELE.

## NOTICES OF BOOKS

*Baltimore Lectures.* By Lord KELVIN. London: C. J. Clay and Sons. Pp. 694.

THIS volume is based upon the course of twenty lectures delivered by Lord Kelvin (Sir W. Thompson) in 1884 at the Johns Hopkins University, his subject being "Molecular Dynamics and the Wave Theory of Light." Nearly half the volume is occupied by twelve appendices on "Allied Subjects"; and as many of the lectures themselves have been largely re-written, it may be said to contain all that is known of the branches discussed.

We have read the book from cover to cover, hoping to find something easy as a basis for review, but when we remind our readers that the lectures were delivered to an audience of scientific men, many of them of world-wide reputation, and that the assumption of "common knowledge" was pitched at a correspondingly high level, we may be excused for saying we propose to make no comments as to their accuracy and value. The appendices are on a different footing; they have been written at different periods and for different audiences, and, excepting for the mathematics, they are both interesting and comparatively easy to understand, so much so that in two instances we disagree with Lord Kelvin's conclusions, and venture to set forth the grounds for so doing.

In Appendix B, Lord Kelvin describes some careful experiments he made with regard to the path of a particle in enclosed areas of different shapes, and having summed the kinetic energy along the co-ordinates during several hundred flights, he arrives at the conclusion that the Boltzmann-Maxwell doctrine as to the equality of the energy in all directions is not justified.

It seems to us that the experiment fails for two reasons:—Firstly, as only one particle is considered, the effect of collisions in "averaging" energy is ignored; and, secondly, "several hundred flights" are altogether inadequate to give a true idea of the average direction or motion of a single particle.

Maxwell considered a group of many millions of atoms, and stated that they would behave in a certain way. Lord Kelvin examines a single particle and finds that it does not, in an extremely limited period, harmonise with Maxwell's doctrine. Probably Maxwell would have been astonished if it did.

The Boltzmann-Maxwell doctrine may be wrong; in fact, Lord Kelvin gives other reasons, which appear conclusive, for stating that it is, but it is not obvious how the experiments described have any value.

Appendix D deals with a speculation as to the amount of matter in the universe in an interesting but unconvincing manner. Lord Kelvin says on page 534:—"Hence, if the thousand million suns had been given at rest twenty-five million years ago, uniformly distributed throughout the supposed sphere (of  $3.09 \times 10^{16}$  kilometres radius), many of them would now have velocities of 20 or 30 kilometres per second, while some would have less, and some probably greater velocities than 108 kilometres per second; or, if they had been given thousands of millions of years ago at

rest so distributed that now they were equally spaced throughout the supposed sphere, their mean velocity would now be about 50 kilometres per second. This is not unlike the measured velocities of stars, and hence it seems probable that there might be as much matter as one thousand million suns within the distance  $3.09 \times 10^{16}$  kilometres.

Surely, in either of the assumed cases, the paths of all the stars would be sensibly centripetal, which the observed paths of the few stars recognised to be in motion is not; this consideration alone appears to put the assumed "initial state" out of court. Again, the visible stars are not by any means even approximately uniformly distributed in space, and it is highly probable that they have not been so, so recently as twenty-five million years ago; whether they might have been during the longer but vaguer period of "thousands of millions of years ago" is another question, but if they were, it is reasonably certain, from their present distribution and the paths of such of them as are approximately known, that they were not then at rest. Again, considering the few stars whose parallaxes have been measured, and the uncertainty attaching to the values of the parallaxes and proper motion, it is taking full advantage of the figures to say "this is not unlike the measured velocities of stars."

So it seems that if one thousand million suns had been distributed a certain long time ago in a manner in which they were not, or if they had been an uncertain time ago so distributed, that they would now be distributed in a manner in which they are not, their mean velocities would be approximately equal to the assumed mean velocities of the visible stars, though their directions would be different. "Hence it seems probable that there might be as much matter as one thousand million suns within the distance  $3.09 \times 10^{16}$  kilometres." Well, there may be; but there is not much reliable evidence either way.

*The Electrolysis of Water; Processes and Applications.* By VIKTOR ENGLEHARDT. Authorised English Translation by J. W. RICHARDS, M.A., &c. With 90 Figures and 15 Tables in the Text. Easton, Pa.: The Chemical Publishing Company. 1904. Pp. 140.

THIS volume is the first of a collection of monographs of applied electrochemistry, and is devoted to a description of the processes used for the electrolysis of water.

The different processes for effecting this decomposition are divided into three principal groups:—

- A. Processes and apparatus for the separate production of oxygen and hydrogen. Sub-divided into—(a) With porous diaphragms of non-conducting material; (b), with complete non-conducting partitions; and (c), with complete or perforated conducting partitions.
- B. Processes and apparatus for the electrolysis of water without separation of the gases (production of detonating gas).
- C. Processes for the simple evolution of oxygen—(a) Through depolarisation at the cathode; and (b) by the precipitation of metal at the cathode.

All the above processes are fully described in the third and most important section, at the end of which is a table giving them all in chronological order.

In the above section, and from the applications described in it, it may be assumed that the question of the industrial electrolysis of water for the purpose of obtaining oxygen and hydrogen has been solved in numerous ways, but the applicability of these processes has become much more of a practical question in these days of cheap power. In Section IV. the author describes various installations and their capacity of competing commercially with other methods, and he gives in compact form the cost of plant and cost of working of these different processes.

Special applications of the uses of oxygen and hydrogen, and the mixed gases, are described at the conclusion of the book, and some new propositions concerning their use are alluded to at length.

Besides forming a valuable collection of processes for

the production of oxygen and hydrogen by the electrolysis of water, this volume is rich in suggestions of ways and means for overcoming difficulties in electrochemical operations in general, and will be found of value to electrochemists engaged in other branches of the subject.

The book closes with an index of author's names, which, though useful to those who are very well acquainted with the subject, by no means makes up for the lack of a general index; there is, however, a very full table of contents, and this must serve in its place.

*Practical Chemistry.* Part II. By WILLIAM FRENCH, M.A., F.I.C., and T. H. BOARDMAN, M.A. London: Methuen and Co. 1904. Pp. 126.

PART I. of this book was published as long ago as 1900, and was noticed in this column in May of that year.

Part II., which has recently appeared, continues as far as possible according to the scheme adopted in Part I.

Chapters I. and II. are on the properties of gases, such as the effects of changes of temperature and pressure, and their densities, solubilities, and diffusion. Then follow several chapters on the laws of chemical combination, equivalents, the atomic theory, symbols, &c. Chapter VIII. is on sulphur and its compounds, and deals with this subject in an adequate manner, while Chapters IX. on nitrogen and X. on carbon, deal in a similar manner with some of the compounds of these elements.

The illustrations are superior to those so often met with in this class of book, and we are glad to find the authors have risen superior to the well known picture of a wash-bottle, a funnel, or a piece of platinum wire. We must, however, call attention to the inconvenient arrangement of the table of contents; the page numbers of Chapters V. and VII. are not given at all, and we find that the pages given in the other cases do not refer to the page on which the chapters commence, but to certain paragraphs in these chapters. Still, this is only a small matter, and does not detract from the value of the book as an educational work.

*Calculating Tables.* By Dr. H. ZIMMERMANN. London: Asher and Co.

THIS work contains a multiplication table of all numbers to 1000 by 1 to 100, and various tables of squares, cubes, square and cube roots, functions of  $\pi$ , and other quantities which are continually being used in calculations; the object being, as stated in the preface, to avoid the use of logarithms as much as possible. The practical value may be gauged by the fact that the preface is dated Berlin, 1889, and after the experience of fifteen years it has been considered a judicious speculation to publish an English edition.

The compiler of the tables is so confident of their accuracy that he has offered a reward to any user of the tables who may detect any errors in them. As this challenge is now fifteen years old, we may conclude that their claim to accuracy is vindicated.

As an adjunct to the usual tables of logarithms these tables should prove useful to many persons.

*Introduction to Physical Chemistry.* By JAMES WALKER, D.Sc., &c. London: Macmillan and Co. Pp. 364.

THIS book, originally published in 1899, has now reached its third edition, which is fair evidence of its value.

It is clearly written throughout, and the mathematical expressions are kept to a minimum, such as should be within the capacity of every student to understand.

A valuable feature of the work is the system of references at the end of most chapters to the original papers on the latest development of the subject matter treated of. Many of these references are to papers published in 1903; so it may be that the author has done as much as is possible to keep his work abreast of the times.

The work is worthy of a place on the shelves of every chemist's library.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. xxxviii., No. 13, March 28, 1904.

**Certain Physical Constants of Phosphorus Fluorides.**—Henri Moissan.—The determination of the melting- and boiling-points of the various fluorides of phosphorus presents certain difficulties, on account of their easy decomposition by water. In the preliminary experiments the author tried to determine these constants by means of a thermometer, but the necessity of operating on a very small quantity of material and in a perfectly closed vessel rendered the equilibrium of temperature between the liquid and the thermometer doubtful. His determinations were finally made by means of a thermo-electric cell, and the melting- and boiling-points of the three fluorides of phosphorus, under normal pressure, are shown by the following table:—

|                            | Melting-point. | Boiling-point. |
|----------------------------|----------------|----------------|
| PF <sub>3</sub> .. .. .    | -160°          | -95°           |
| PF <sub>5</sub> .. .. .    | -83°           | -75°           |
| PF <sub>3</sub> .O .. .. . | -68°           | -40°           |

**Separation of Chromium and Vanadium.**—Paul Nicolardot.—Already inserted in full.

**Preparation of Ether Oxides by means of Magnesium Compounds and Halogen Methylic Ethers, X.CH<sub>2</sub>OR.**—J. Hamonet.—M. Henry used the action of halogen methylic ethers, X.CH<sub>2</sub>OR, on organo-zinc compounds to show that, by means of successive substitutions, methylic alcohol can be transformed into all its superior homologues: (CH<sub>3</sub>)<sub>2</sub>Zn + 2XCH<sub>2</sub>OR = ZnX<sub>2</sub> + 2CH<sub>3</sub>CH<sub>2</sub>OR. The author finds that this reaction applies equally well to M. Grignard's magnesium compounds, R.MgX + XCH<sub>2</sub>OR = MgX<sub>2</sub> + R.CH<sub>2</sub>OR. By applying this method, the author prepares amyloxypropylic ether, C<sub>5</sub>H<sub>11</sub>OC<sub>3</sub>H<sub>7</sub>, boiling at 130°; benzyloxyethylic ether, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>OCH<sub>3</sub>, boiling at 170°; and phenylethyloxy-methylic ether, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>CH<sub>2</sub>OCH<sub>3</sub>, a liquid never before obtained, boiling at 189–190°.

**Phospho-nitrated Bases of the Type (RNH)<sub>3</sub>P=NR.**—P. Lemoult.—In a previous investigation the author proved the existence of a new phosphorus base derived from aniline:—Trianilidophenylphosphimide, (C<sub>6</sub>H<sub>5</sub>NH)<sub>3</sub>P=NC<sub>6</sub>H<sub>5</sub>. He now prepares certain homologous amines of aniline, particularly the *o*-toluidine and the *as-m*-xylylidine. The salts derived from these two series can be considered as derived from an analogous monacid base into an anilide base of the general type (RNH)<sub>3</sub>P=NR (R = C<sub>6</sub>H<sub>5</sub> or C<sub>7</sub>H<sub>7</sub> or C<sub>8</sub>H<sub>9</sub>), becoming a salt in the manner of ammonia by simply fixing the elements of a molecule of monobasic acid. It appears that the basic activity and the stability of the base itself diminish as it rises in the series.

**Application of Acetylene Gas to the Heating of Germination Stoves by means of an Automatic Regulator of Temperature.**—H. Joffrin.—The author describes a germination stove which can be used with acetylene gas when ordinary coal-gas is not attainable. A drawing is given of the apparatus.

## MISCELLANEOUS.

**Protection of Steel from Corrosion.**—Experiments have been made recently by Prof. C. L. Norton, of the Insurance Engineering Experiment Station, Boston, U.S.A., on the best methods of protecting steel used in buildings from corrosion. In some of the high buildings in America the question of the corrosion of the steel

footings is one of the utmost importance, when embedded in damp ground. Under such conditions the steel may be corroded by electrolysis from leaking currents from an electric rail or tramway, though dry steel will carry electric currents without injury. It has been found that Portland cement concrete affords perfect protection to steel embedded in it. The specimens of steel were cut to about 3 inches by 1 inch, and were of all thicknesses from 1/50 inch to 1 1/4 inch. Some were cut dry, some in oil, some in water, so that all conditions met with in regular practice should be included. Each piece was weighed and calipered at several points, and incorporated in a block or brick of concrete sufficiently large to cover it everywhere to a depth of one inch and a-half. The blocks were divided into three groups, one group being set out of doors, one stored in a damp and odorous cellar, and the third being treated in the "corroders," or steam and carbon dioxide tanks. Other specimens were further exposed in tide water, in sewers, and over furnaces with constant contact with furnace gases. After varying lapses of time, from one to nine months, the specimens were broken out of the briquettes, cleaned, weighed, and calipered. Not one specimen showed any sensible change in weight or dimensions except where the cement had been poorly applied.

**The Precipitation of Colloidal Solutions of Sulphide of Arsenic.**—F. W. Kuster and G. Dahmer.—In a preceding communication the authors have shown that sulphuretted hydrogen reacts quantitatively on aqueous solutions of arsenious acid, transforming them into sulphide, the sulphide formed remaining for a longer or shorter time in solution in the colloidal state (*Zeit. Anorg. Chem.*, vol. xxxiii., p. 105). This colloidal solution is precipitated very rapidly by coarsely ground Iceland spar. The other substances tried, such as heavy spar, precipitated sulphate of baryta, wood-charcoal, oxide of copper, and powdered glass, are much less active. The precipitation is further considerably facilitated by agitation and by the use of a large excess of the precipitating agent. The use of heavy spar (or of the other substances tried) does not seem, as mentioned by Vanino (*Ber.*, vol. xxxv., p. 662), to show a distinction between true solutions and colloidal solutions; for as we should preferably operate in the presence of a considerable quantity of the precipitating agent (100 parts of this latter to 1 part of the colloid to be precipitated, for example), it is to be feared that the materials really in solution are only more or less precipitated by reason of surface absorption.—*Zeit. Anorg. Chem.*, vol. xxxiv., p. 410.

## MEETINGS FOR THE WEEK.

- MONDAY, May 2nd.**—Royal Institution, 5. Annual Meeting.  
— Society of Arts, 4.30. (Cantor Lectures). "The Majolica and Glazed Earthenware of Tuscany," by Prof. R. Langton Douglas, M.A.  
— Society of Chemical Industry, 8. "Determination of Minute Quantities of Bismuth in Copper Ores" and "Determination of Minute Quantities of Arsenic in Copper Ores and Metallurgical Products," by T. C. Cloud. "Testing Colloids," by Prof. E. J. Mills and A. Gray.  
**TUESDAY, 3rd.**—Royal Institution, 5. "Meteorites," by L. Fletcher, F.R.S., &c.  
— Society of Arts, 4.30. "Canada and Great Britain," by W. L. Griffith.  
**WEDNESDAY, 4th.**—Society of Arts, 8. "Statistics of the World's Iron and Steel Industries," by William Pollard Digby.  
— Society of Public Analysts, 8. "Cod-liver and other Fish Oils" and "Note on Mushroom Ketchup," by J. F. Liversidge. "Note on some Constants obtained in the Examination of Margarine," by Edward Russell and V. H. Kirkham. "Note on the Estimation of Sugars in Concentrated Malt Extract," by A. R. Ling and Theodore Rendle.  
**THURSDAY, 5th.**—Royal Institution, 5. "Great Britain and Europe (1763–1793)," by Arthur Hassall, M.A.  
— Chemical, 8. "Slow Combustion of Ethane," by W. A. Bone and W. E. Stockings. "Note on

the Hydrolysis of Starch by Diastase," by J. S. Ford. "The Resin Acids of the *Coniferae*—Part I. The Constitution of Abietic Acid," by T. H. Masterfield and G. Bagby. "The Action of Radium Rays on the Halides of the Alkali Metals and Analogous Effects produced by Heat," by W. Ackroyd. "The Dynamic Isomerism of Glucose and of Galactose—Solubility as a means of Determining the Proportions of Dynamic Isomerism in Equilibrium," by T. M. Lowry. "A Study of the Substitution Products of *ar*-Tetrahydro-*a*-naphthylamine, *ar*-4-bromotetrahydro-*a*-naphthylamine, and *ar*-Tetrahydro-*a*-naphthylamine-4-sulphonic Acid," by G. T. Morgan, Miss F. M. G. Micklethwait, and H. B. Winfield. "The Additive Products of Benzylideneaniline with Methylacetoacetic Ester and Acetoacetic Ester," by F. E. Francis and Miss M. Taylor.

FRIDAY, 6th.—Royal Institution, 9. "Anthropoid Apes," by P. Chalmers Mitchell, D.Sc.

Physical, 8. "An Experiment with Lubricating Oil," by W. A. Price. "Some Instruments for the Measurement of Large and Small Alternating Currents," by W. Duddell. Exhibition of Apparatus from the National Physical Laboratories.

SATURDAY, 7th.—Royal Institution, 8. "Sonata Style and the Sonata Forms" (with Musical Illustrations), by Donald Francis Tovey, B.A.

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## CONTENTS.

| ARTICLES:—                                                                                                                                | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------|------|
| Physical Constants at Low Temperatures, by Prof. James Dewar, M.A., F.R.S., &c.                                                           | 217  |
| Preparation and Properties of Pure Colloidal Silver, by A. Chassevant                                                                     | 218  |
| New Method for the Estimation of Organic Matters in Water, more particularly in Waters containing Chlorides and Bromides, by C. Lenormand | 219  |
| PROCEEDINGS OF SOCIETIES—                                                                                                                 |      |
| ROYAL INSTITUTION—Annual General Meeting                                                                                                  | 220  |
| CHEMICAL SOCIETY.—Elements and Compounds—Vapour Density of Hydrazine Hydrate—Combining Volumes of Carbon Monoxide and Oxygen—&c.          | 220  |
| NOTICES OF BOOKS                                                                                                                          | 225  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                     | 226  |
| MISCELLANEOUS                                                                                                                             | 227  |
| MEETINGS FOR THE WEEK                                                                                                                     | 227  |

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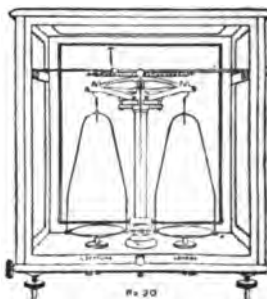
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2319.

## PHYSICAL CONSTANTS AT LOW TEMPERATURES.\*

### I. THE DENSITIES OF SOLID OXYGEN, NITROGEN, HYDROGEN, &c.

By Prof. JAMES DEWAR, M.A., LL.D., D.Sc., F.R.S.

(Concluded from p. 207).

5. With these values for the molecular volumes at absolute zero of oxygen, nitrogen, and hydrogen, namely, 21.21, 25.49, and 24.18 respectively, and knowing that the molecular volume of ice at absolute zero is 19.21 and of carbonic acid is 25.7 as deduced from a study of the coefficients of expansion at low temperatures, we can find the contraction or expansion on the assumed hypothetical production of these compounds from their elements at the zero of temperature ("Coefficients of the Cubical Expansion of Ice, Hydrated Salts, Soluble Carbonic Acid, and other Substances at Low Temperatures," *Roy. Soc. Proc.*, vol. lxxiii.).

Thus 100 volumes of mixed solid hydrogen and oxygen become, after combination, 55.22 volumes of ice, or there is a contraction of 45 per cent. This is of the same order of magnitude as for  $N_2O$  and  $Li_2O$ , whose contraction from solid oxygen, solid nitrogen, and metal amounts to 60 per cent. On the other hand, the production of carbonic acid from diamond ( $V_0 = 6.82$ ) or graphite ( $V_0 = 10.44$ ) and solid oxygen gives in the former case a slight expansion of 4½ per cent, but in the latter case a slight contraction of 3 per cent.

The case of carbonic oxide ( $V_0 = 24.5$ ) is interesting; produced from diamond and oxygen it expands by 74½ per cent, and from graphite and oxygen it expands by 54½ per cent, but on the addition of another atom of oxygen the resulting product, carbonic acid, undergoes a contraction of 27 per cent.

6. Berthelot made a minute examination of the values of  $d(p)/dp$  in reduced co-ordinates for eight substances whose critical temperatures range from that of carbonic acid to that of hydrogen; and came to the conclusion that the co-volume is one quarter of the critical volume, at least for low pressures ("Sur les Thermomètres à Gaz. Trav., et Mém. Poids et Mesures," vol. xiii.).

Guldberg (*loc. cit.*) from a careful graphical discussion of Young's results, arrived at the conclusion that the critical volume is 3.55 times the volume at absolute zero.

An independent examination of the reduced curve for Young's fluorobenzene, showed that the reduced volume for a reduced pressure between 0.05 and 0.10 is very approximately 0.4; in other words, that the critical volume is two and a-half times the co-volume. And it is to be noted that the variations of the reduced volume in this neighbourhood are very slight compared with the change of the reduced pressure. Further, this range of reduced pressure will cover the extent of the present experiments whatever may eventually prove to be the value of the real critical pressure. My old experiments gave as a maximum a critical pressure of a little over 15 atmospheres.

Hence taking Van der Waals's  $b = 12$  for hydrogen, these results give respectively 48 c.c., 42.6 c.c., and 30 c.c. as the critical volume, with the corresponding critical densities, 0.0208, 0.0235, and 0.033.

What further considerations can be brought to determine between these results? Van der Waals gives, for corre-

sponding states of two bodies, the equation ("Continuity, &c.," English translation, p. 491)—

$$\frac{d}{d'} = \frac{M}{M'} \frac{p_c}{p'_c} \frac{t'_c}{t_c},$$

where  $d$  is density and  $M$  is molecular weight. Now in the liquid states for moderate pressure, the density changes but slightly, hence we may assume with very little error that the boiling-point densities belong to corresponding states. Comparing, therefore, hydrogen with oxygen, we get—

$$\frac{0.070}{1.138} = \frac{2}{32} \frac{p_c}{t_c} \frac{155}{50} \text{ or } \frac{t_c}{p_c} = 3.15;$$

and comparing hydrogen with nitrogen we have—

$$\frac{0.07}{0.79} = \frac{2}{28} \frac{p_c}{t_c} \frac{127}{35} \text{ or } \frac{t_c}{p_c} = 2.93;$$

hence, taking the mean, we have for hydrogen  $t_c/p_c = 3.04$ . Now, Van der Waals's theory makes  $p_c v_c/t_c = \frac{8}{3} R$ , but experiment seems to require  $p_c v_c/t_c$  to be equal to  $\frac{1}{3.77} R$ ,

and when  $p$  is measured in atmospheres,  $R = 41.0183$  for hydrogen, for which, therefore,—

$$\frac{p_c v_c}{t_c} = \frac{41.0183}{3.77} = 10.8801.$$

This gives  $v_c = \frac{10.8801}{3.0} = 3.508$ , and the critical density

as 0.0285, a result midway between that got from Guldberg's ratio and that derived from Young's reduced fluor-benzene.

Further experimental results can be brought to bear on the question. I have recently measured directly the heat of vaporisation of liquid hydrogen at the boiling-point, and find it to be not more than 125 calories per gram. Assuming the Rankine equation  $\log_{10} p = A - B/t$ , the molecular heat of vaporisation is  $2B \log_e 10$ . Hence we get  $B = 54.34783$ ; and determining  $A$  from the values of  $p$  and  $t$  at the boiling-point we have in atmospheres,—

$$\log_{10} p = 54.34783 \left( \frac{1}{20.5} - \frac{1}{t} \right).$$

Now, at the critical point we have found  $t_c = 3.04 p_c$ , hence combining these results we have finally,—

$$t_c = 33.88^\circ, \text{ and thence } p_c = 11.145 \text{ atmospheres.}$$

Before leaving these results we may write the Rankine equation in Van der Waals's form, namely—

$$-\log \epsilon = \frac{54.34783}{t_c} \frac{1-m}{m},$$

where  $\epsilon$  and  $m$  are the reduced pressure and temperature, and Van der Waals's  $f$  is  $54.34783/t_c$ , or with the above result 1.604, about half the usual value of  $f$ . In like manner Trouton's constant, namely, the ratio of the molecular latent heat to the absolute temperature of the boiling-point, is  $125/20.5$  or 6.096, again only about half the usual value.

In this connection we may refer to Olszewski's experimental observation of the temperature, 192° absolute, at which the Joule-Thomson effect vanishes when the expansion is from a great pressure, in this instance between 110 and 117 atmospheres. With the usual Van der Waals's notation we may express the connection between this inversion temperature and the critical temperature in either of the forms,—

$$t = \frac{27}{4} \left( \frac{v-b}{v} \right)^2 \cdot t_c \dots \dots \dots (9),$$

or—

$$t = 3 \left( 1 + \frac{1}{2} \sqrt{1 - \frac{p}{9p_c}} \right)^2 \cdot t_c \dots \dots (10),$$

the former equation shows that for small pressures and consequent values of  $v$  so great that  $b$  may be neglected

\* A Paper read before the Royal Society, March 17, 1904.

in comparison with  $v$ , the critical temperature is  $4/27$  of the temperature of inversion. But when the pressure is great, the ratio  $b/v$  cannot be neglected, and the critical temperature is a greater multiple of the temperature of inversion. The second of these equations shows that the initial pressure must not exceed nine times the critical pressure. Assuming a critical pressure of 16 atmospheres for hydrogen, and taking  $p = 117$  atmospheres and  $t = 192^\circ$ , this equation gives the critical temperature as  $42^\circ$  absolute. Again, for  $p_c = 15$  atmospheres,  $t_c = 46^\circ$  and for  $t_c = 32^\circ$ ,  $p_c = 41$  atmospheres.

Results derived from a discussion of similar equations depending on Clausius's formula, Berthelot's "modified" Van der Waals's, or Reinganum's formula, are still farther from the value we expect.

In part explanation of this failure it is to be noted that these formulæ are but the best theoretical approximations we have at hand, and while they are useful within short ranges we can hardly expect the same accuracy over a temperature range of five or six times the critical temperature.

Again, for a very large number of bodies the product of  $\alpha$ , the co-efficient of expansion at the boiling-point, and the critical temperature is constant and about 0.6 to 0.7.

Thus for oxygen, from equation (2), we have  $at_c = 0.61$ .

For nitrogen we get  $at_c = \frac{0.0750}{0.8042 \times 15} \times 127 = 0.79$ , but

for hydrogen we have  $\frac{0.0054}{0.07 \times 5.8} \times 34 = 0.45$ , and even if we take the critical temperature as high as  $42$ ,  $at_c$  only reaches 0.56.

7. There are, therefore, as far as we can see at present, and as far as theoretical considerations can aid us, great departures shown by hydrogen from what are fairly general results. Van der Waals's  $f$  and Trouton's constant are each only about half the usual values; and we have now found, from the consideration of the point of inversion of the Joule-Thomson effect, and of the product  $at_c$ , variations greater than the average from the values we should have expected. Further experiment will be necessary before these discrepancies can be cleared up.

## PREPARATION AND PROPERTIES OF PURE COLLOIDAL SILVER.

By A. CHASSEVANT.

IN these new experiments I prepared colloidal silver according to Schneider's method (*Berichte*, vol. xxv., p. 1281) by mixing 500 c.c. of a 10 per cent solution of nitrate of silver, 500 c.c. of a 30 per cent solution of ferrous sulphate, and 700 c.c. of a solution containing 280 grms. of crystallised citrate of soda.

A reddish deposit is obtained, which is separated from its ferruginous mother-liquor, by means of a filter-pump, after having stood for half-an-hour.

The precipitate is re-dissolved in a stream of distilled water, and the solution is treated with about twice its volume of alcohol at  $95^\circ$ . The addition of the alcohol causes a bluish-black deposit to be formed, a precipitate of *sol*. The mixture is filtered through a Chamberland cylinder. The alcoholic solution of ferric citrate traverses the filter, and is collected in a recipient *ad hoc*; the precipitate of colloidal silver is deposited on the outside of the filter in the form of a pasty crust; after some time the filter no longer acts; it is then withdrawn from the liquid, but the aspiration is continued until the crust assumes an iridescent, brilliant metallic appearance, and seems to be dry.

Then placing the filter in a glass containing distilled water the *sol* is allowed to re-dissolve.

I have observed that the Chamberland cylinder, the pores of which are obstructed by the colloidal molecules of silver, acts as a dialyser, and that on adding water and applying the vacuum we can cause the ferric salts and other im-

purities to pass through into the interior, the *sol* of colloidal silver remaining in the outer solution.

The solution may be again precipitated by alcohol at  $95^\circ$ , and again purified, as has just been described. If we have been careful to avoid the presence of all crystalloids, and especially of mineral acids, these operations can be repeated several times, and the colloidal silver retains its properties of a *sol*. Solutions of pure colloidal silver have no longer any tendency to become precipitated as a *gel*. Thus, while in the presence of the mother-liquor during its preparation the black insoluble *gel* is produced in a few hours, solutions of colloidal silver free from electrolytes can be kept for several weeks without any change.

It is interesting, in practice, to notice the action of the alcohol; when we are obliged to leave the colloidal silver in very impure circumstances, it is sufficient to cause the precipitation of the *sol* by means of alcohol; the colloidal silver thus precipitated retains its properties of a colloid for a very long time, even in the presence of an excess of crystalloid salts, which under ordinary circumstances would rapidly transform the colloidal silver into the insoluble *gel*.

By applying this method of purification, I have succeeded in obtaining, after a first purification, a solution of colloidal silver containing per 100 c.c. :—0.480 grm. of material dried at  $100^\circ$ , 0.478 grm. of material stable at a bright red heat, and 0.383 grm. of silver, that is 81.7 per cent of silver; the difference was almost entirely iron.

After two purifications, 100 c.c. of the solution contained 0.300 grm. of material dried at  $100^\circ$ , and 0.2897 grm. of silver, or 96.59 per cent of silver.

This pure solution of colloidal silver has all the properties of colloids, which have been published by my friend Posternak and myself, and which have been partly observed by others.

The various electrolytes, dilute carbonate of soda, nitrate of potassium, perchloride of iron, sulphate of copper, &c., cause the precipitation of the *gel*.

If we evaporate the solution *in vacuo*, and dry the pasty mass collected on a Chamberland filter, metallic silver is formed. On the other hand, the aqueous solution of the *sol* is very stable. We can even keep this precipitated *sol* on porcelain in concentrated alcohol. Colloidal silver thus purified dissolves in alcohol, but only in the presence of water; it is insoluble in absolute alcohol. Purified colloidal silver dissolves easily in glycerin at  $30^\circ$ . We obtain organosols observed by Schneider, which will be the subject of a future paper.

If we treat a solution of pure colloidal silver with another colloid, such as gum, gelatin, or a sodic solution of acid-albumose, we can obtain, by evaporating in the cold *in vacuo*, a solid mass in which the silver has preserved its physical colloidal state, and can be re-dissolved in distilled water. I shall return to this in a future paper on these preparations.

The summary of all these facts makes us admit with Carey Lea (*Zeit. Anorg. Chem.* vol. xiv., p. 341) that all the preparations of colloidal silver contain the silver, not in a special allotropic form, but that the fine particles of silver, which were unable to become conglomerated at the moment of their production, have remained in the free state in solution.

The transformation of the *sol* into the *gel*, and into metallic silver, takes place naturally every time the physical molecules are condensed or brought into contact one with the other, either under the influence of electrolytes, by precipitation, by crystallisable salts, or by reason of the loss of liquid support through dilution; by the evaporation of solutions of pure colloidal silver, or by simple mechanical action, as we have shown by grinding up colloidal silver. On the other hand, the colloidal form of silver is stable in pure solution in water, alcohol, or glycerin. In the presence of other colloids we can again conserve silver in the colloidal form, even in the solid state, as shown by Paal (*Berichte*, vol. xxxv., p. 2224), with lysoalbumic acid.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 1.

NEW METHOD FOR THE  
ESTIMATION OF ORGANIC MATTERS IN WATER,  
MORE PARTICULARLY IN WATERS  
CONTAINING CHLORIDES AND BROMIDES.

By C. LENORMAND.

WHEN we endeavour to estimate organic matter, by the ordinary methods, in waters containing alkaline chlorides and bromides—such as sea-water, for example,—we obtain results that are necessarily erroneous; in fact, chlorine and bromine are given off as soon as we add the sulphuric acid to the manganic solution, and thus, through this action, the amount of organic matter found is too high.

Among the methods which may be recommended for preventing this error we will describe one which has given us the best results, and which is based on a new adaptation of Duboscq's colorimeter. We have used it for estimating the organic matter in sea-water, and its use can be advantageously extended to the estimation of the organic matter in fresh waters by introducing a modification which we will describe at the end of this paper. We propose to examine successively its technique, its constancy, and its sensitiveness. To begin with, the principle adopted is as follows:—

Boil the water under analysis with a titrated alkaline solution of permanganate of potassium. From this we deduce, by comparison in the colorimeter with the original titrated solution, the amount of permanganate of potash used; that is to say, the quantity of oxygen absorbed by the organic matter.

I. The solutions necessary for the estimation of the organic matter by this new method are—

1. A solution containing 0.395 grm. of permanganate of potassium per litre (10 c.c. of this solution corresponds to 1 m.grm. of oxygen).
2. A saturated solution of bicarbonate of soda.

Place 100 c.c. of the water to be examined in a beaker with 10 c.c. of the permanganate and 10 c.c. of the bicarbonate of soda solutions. Boil for ten minutes, and, after cooling, bring the volume up to 100 c.c. with distilled water. Allow to settle completely, and decant the liquor into one of the colorimeter glasses, and in the other place a solution formed of 10 c.c. of the manganic liquor and 90 c.c. of distilled water. Then make the tints appear equal (see remark at the end of the paper).

Now, let  $x$  be the quantity of permanganate remaining after boiling,  $p$  the amount of permanganate in 100 c.c. of the check sample,  $H_1$  the degree under which the water in question is being examined, and  $H$  that of the check sample. We get—

$$x = p \frac{H_1}{H};$$

or, replacing  $p$  by its value,—

$$x = 0.00395 \frac{H}{H_1},$$

the amount of permanganate that has disappeared during boiling will be expressed by the formula—

$$0.00395 - 0.00395 \frac{H}{H_1}, \text{ or } 0.00395 \left( \frac{H_1 - H}{H_1} \right),$$

but 0.00395 of permanganate of potassium corresponds to 1 m.grm. of oxygen; the quantity  $0.00395 \left( \frac{H_1 - H}{H_1} \right)$  will correspond to a quantity equal to  $\frac{H_1 - H}{H_1}$ .

*Example.*—Let  $H_1 = 40$  and  $H = 26.4$ , the quantity of oxygen fixed by 100 c.c. of water will be  $\frac{40 - 26.4}{40} = 0.34$  m.grm., or 3.4 m.grms. per litre.

II. The method is constant in its applications, and the intensity of the final tint obtained always gives the same results with one particular sample, no matter how many

times it is repeated, provided the operations are conducted under identical conditions.

The following table shows the results obtained in three series of experiments carried out successively on one sample of sea-water diluted with distilled water.

We see that, within one-tenth of a milligram., the results obtained are the same, and that the variations between the three analyses are not greater than two or three hundredths of a milligram. of oxygen on the total amount of organic matter contained in a litre of water.

*Dilute Sea-Water.*

| $H_1$ .                  | H.   | $\frac{H_1 - H}{H_1}$ . |
|--------------------------|------|-------------------------|
| <i>First Experiment.</i> |      |                         |
| 30                       | 23.5 | 0.216                   |
| 30                       | 23.7 | 0.210                   |
| 30                       | 23.6 | 0.213                   |
| 30                       | 23.5 | 0.216                   |
| 30                       | 23.7 | 0.210                   |
| Mean .. ..               |      | 0.213                   |

Oxygen fixed per litre .. .. 2.13 m.grms.

*Second Experiment.*

| $H_1$ .    | H.   | $\frac{H_1 - H}{H_1}$ . |
|------------|------|-------------------------|
| 30         | 23.5 | 0.216                   |
| 30         | 23.6 | 0.213                   |
| 30         | 23.7 | 0.210                   |
| 30         | 23.7 | 0.210                   |
| 30         | 23.6 | 0.213                   |
| Mean .. .. |      | 0.212                   |

Oxygen fixed per litre .. .. 2.12 m.grms.

*Third Experiment.*

| $H_1$ .    | H.   | $\frac{H_1 - H}{H_1}$ . |
|------------|------|-------------------------|
| 30         | 23.6 | 0.213                   |
| 30         | 23.4 | 0.220                   |
| 30         | 23.6 | 0.213                   |
| 30         | 23.5 | 0.216                   |
| 30         | 23.5 | 0.216                   |
| Mean .. .. |      | 0.215                   |

Oxygen fixed per litre .. .. 2.15 m.grms.

III. This method is also very sensitive; its sensitiveness is certainly as great as that of the method in general use for the estimation of organic matter in fresh waters.

In fact, if to a sample of sea-water we add 2, 4, 6, 8, or 10 c.c. of a solution of peptone containing 0.1 m.grm. of this substance per c.c., we obtain figures between which the differences are practically constant, as will be seen by the following table:—

| Peptone added to sample of sea-water. | $H_1$ . | H.   | Differences, in m.m. | Oxygen fixed per litre, in m.grms. | Differences, in m.grms. |
|---------------------------------------|---------|------|----------------------|------------------------------------|-------------------------|
| 0.0                                   | 40      | 29.9 |                      | 2.525                              |                         |
| 0.2                                   | 40      | 26.9 | 3.0                  | 3.275                              | 0.750                   |
| 0.4                                   | 40      | 24.0 | 2.9                  | 4.000                              | 0.725                   |
| 0.6                                   | 40      | 21.1 | 2.9                  | 4.725                              | 0.725                   |
| 0.8                                   | 40      | 18.2 | 2.9                  | 5.450                              | 0.725                   |
| 1.0                                   | 40      | 15.2 | 3.0                  | 6.200                              | 0.750                   |

This table shows further that for a difference of 2.9 m.m. between two successive values of  $H$ , there is a difference of 0.725 m.grm., or one thousandth, in the corresponding values of the oxygen absorbed, a variation of  $\frac{0.725}{2.9} = 0.25$  m.grm., and for 1/10th of a thousandth, 0.025 m.grm. of oxygen.

Thus an error in reading of  $1/10$ th of a m.m. makes a variation in the amount of oxygen of 0.025 m.grm. more or less.

Now, as with a practiced eye the error of reading can be reduced to its minimum by taking the mean of five or six readings, which by the way can be done very rapidly, we can safely say that the error in the determination of the organic matter contained in a litre of water would be at the most 0.05 m.grm. of oxygen; that is to say, a quantity that may be looked upon as insignificant.

*Application of this Method to the Estimation of the Organic Matter in Fresh Waters.*

In the estimation of the organic matter in sea-water, the clarification of the liquor, before the colorimetric examination, takes place in a few minutes, thanks to the entanglement of the oxide of manganese by the carbonate of magnesium produced during the boiling. With fresh waters, however, this is not the case; the solution maintains for some hours a dull appearance, which renders the colorimetric examination impossible.

But we can make this examination as rapid as that of sea-water by adding, with the other ingredients, 1 c.c. of a saturated solution of sulphate of magnesium; thus the hydrocarbonate of magnesium is formed as in the former case, and the clarification is complete in about ten minutes, or a quarter of an hour at the most.

*Remarks.*—In practice, and with a view to diminishing the errors of observation, it is as well to examine the waters through as thick a layer as possible. But though it is easy to obtain equality of tints with slightly coloured solutions, the same is not the case when the manganic solution retains almost the whole of its original colour. Under these conditions it is more difficult to arrive at a sensitive tint; but this difficulty can be avoided easily by placing green glasses, with which the apparatus is provided, between the reflecting prisms and the glass cylinders in the slots provided. This new sensitive tint, which is a greenish-yellow, becomes very easy to match.

Finally, we may add that though in particular cases the colour of the check solution is not always absolutely comparable with that of the solution of the water under analysis, it is still easy to make a rigorous comparison. For this purpose we have only to prepare a check solution under the same conditions as that of the water in question, that is to say, boil 100 c.c. of distilled water with 10 c.c. of permanganate of potash, 10 c.c. of bicarbonate of soda, and 1 c.c. of sulphate of magnesia; but the preparation of such a check solution is generally unnecessary, as we have been able to prove.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 15.

## PROCEEDINGS OF SOCIETIES.

### ROYAL INSTITUTION.

*Annual Meeting, May 2nd, 1904.*

Sir JAMES CRICHTON-BROWNE, M.D., Treasurer and Vice-President in the Chair.

THE Annual Report of the Committee of Visitors for the year 1903, testifying to the continued prosperity and efficient management of the Institution, was read and adopted, and the Report on the Davy-Faraday Research Laboratory of the Royal Institution, which accompanied it, was also read. Seventy new Members were elected in 1903.

Sixty-two Lectures and Twenty Evening Discourses were delivered in 1903. The Books and Pamphlets presented in 1903 amounted to about 229 volumes, making with 694 volumes (including periodicals bound) purchased by the Managers, a total of 923 volumes added to the Library in the year.

Thanks were voted to the President, Treasurer, and the Honorary Secretary, to the Committees of Managers and Visitors, and to the Professors, for their valuable services to the Institution during the past year.

The following gentlemen were unanimously elected as Officers for the ensuing year:—

*President*—The Duke of Northumberland.

*Treasurer*—Sir James Crichton-Browne.

*Secretary*—Sir William Crookes.

*Managers*—Dr. Henry E. Armstrong, Sir William Abney, Mr. Shelford Bidwell, Sir Alexander Binnie, Mr. J. H. Balfour Browne, K.C., The Hon. Sir Henry Burton Buckley, Sir Thomas A. De la Rue, Bart., Dr. J. A. Fleming, Sir Victor Horsley, Lord Kelvin, Dr. Ludwig Mond, Sir Owen Roberts, Sir Thomas Henry Sanderson, Sir Felix Semon, and Mr. W. H. Spottiswoode.

*Visitors*—Dr. J. Mitchell Bruce, Mr. J. B. Brown-Morison, Dr. F. Clowes, Dr. Mackenzie Davidson, Mr. W. B. Gibbs, Mr. Francis Fox, Mr. C. E. Melchers, Mr. R. Mond, Mr. J. Callander Ross, The Hon. Walter Rothschild, Mr. Maures Horner, Mr. A. A. Campbell Swinton, Mr. J. J. Vezey, Dr. G. Johnstone Stoney, and Mr. G. P. Willoughby.

### CHEMICAL SOCIETY.

*Extra Meeting, April 19th, 1904.*

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

THIS meeting was held in the Theatre of the Royal Institution, by kind permission of the Managers.

THE PRESIDENT, in opening the proceedings, said:—So many years have elapsed since we enjoyed the delivery of a Faraday Lecture that perhaps a few words of introduction may not be regarded as inopportune. Faraday died in 1867, and immediately after his decease the Council of the Chemical Society met together and determined that, if possible, they would do something permanently to do honour to his memory; and the result was that, after considerable deliberation, they decided to establish this system of lectures. The first of the course was given in 1869 by Dumas, only two years after Faraday's death. Dumas was succeeded by Cannizzaro, Hofmann, Wurtz, Helmholtz, Mendeleeff, and Lord Rayleigh. You will see, therefore, that the most eminent men of science living at the time were ready to accept the invitation to undertake the office of Faraday Lecturer. Owing to accidental circumstances it is, however, a rather long period—some twenty-three years—since we heard the voice of a Faraday Lecturer. Thanks to the kindness of the Members and the Managers of the Royal Institution, the lectures have all, hitherto, been given within this place, the walls of which for so many years echoed to the voice of Faraday, and which were the witness of his teaching and his triumphs. The Council of the Chemical Society were naturally anxious on the present occasion, as on all previous occasions, to find a man who might safely be regarded as qualified by his own researches and by his own contributions to science, worthily to fill the office of Faraday Lecturer; and they are satisfied that they have found such a man among their own Honorary Members. Professor Ostwald is a man who is not only honoured in the country of his adoption, but is famous throughout the world as the apostle of the modern doctrines concerning chemical affinity and the mechanism of chemical action. I will not, however, occupy any further time, but ask Professor Ostwald to be good enough to give us his lecture.

Prof. OSTWALD then delivered the Faraday Lecture, of which the following account is an abstract:—

### Elements and Compounds.

The most general concept underlying all chemical theory is that of a *phase* created by Willard Gibbs. A phase is a physical homogeneous body, which may be either chemi-



cally homogeneous or composed of any number of substances. Every single phase has two degrees of freedom, irrespective of its chemical simplicity or complexity. A difference arises only if a second phase is formed. When that happens, we have two different cases: either the properties of the remaining part of the first phase change during the transformation into a new phase, or they remain constant. Phases of the first order we call *solutions*; of the second, *hylotropic bodies*.

By forming a new phase of a solution, we get two portions possessing different properties. On separating these parts, and on repeating the separation into two phases, every solution finally splits up into a finite number of hylotropic bodies.

Generally, a hylotropic body behaves as such only within certain limits of temperature and pressure. Beyond these limits of stability, it assumes the properties of a solution, and can therefore be split up again into a finite number of other hylotropic bodies. These we call *simpler* bodies than the former. At last it becomes impossible to change the hylotropic body into a solution, because it is not found practicable to reach its limit of stability. *Substances which can form only hylotropic phases we call elements.*

With hylotropic substances, we can again distinguish two cases. (1) Either the hylotropic body can exist as such only at one definite temperature and pressure, and by changing these conditions the body is transformed into a solution. This is the case with solutions of constant boiling-point (for example, the solution of acids investigated by Sir Henry Roscoe) or with cryohydrates, &c. (2) Or there is a finite range of temperatures and pressures within which the phase invariably behaves as a hylotropic body; in this case, we have a *substance* proper. A substance or chemical individual is therefore the limiting case of hylotropic solutions, and is defined experimentally by its not changing its properties if the phase is partially transformed into another, for example, by distillation or crystallisation. This is indeed the way, in which pure substances are defined and identified practically.

The constancy of properties is casually connected with constancy of composition; that is, if it is possible to produce a substance proper from other substances or hylotropic bodies, the ratio of the weights of the component parts must have a certain value in order that this peculiar body of constant properties may be formed. This is the *law of constant proportions*.

In forming such peculiar bodies, elements and compounds behave alike, and there is, therefore, in every case a certain definite relation between a substance and its components, the last being compounds or elements. A compound substance can therefore be split up into, or regenerated from, elements only in one definite manner. If we form a ternary compound ABC of the elements A, B, and C, we get the same substance by forming first AB and uniting it with C, or by first preparing AC and combining this with B, or, lastly, in forming the compound ABC directly from the elements.

By combining A with B, we get a certain ratio of weights between the two elements, and also a ratio for C by combining it with AB to form ABC. Now, as the composition of ABC is independent of the manner of formation, the composition of a substance AC is not more arbitrary, but is already defined by the composition of ABC, for it must contain A and C in the same ratio as in ABC. In the same way, the composition of a substance BC is defined in such a manner that the elements A, B, C, can combine only in definite ratios, regulated by the combining weight of each element for the compound ABC. This is the *law of combining weights*, generally spoken of as the law of atomic weights.

From this law, the *law of multiple proportions* can be deduced easily by applying the same considerations to the case of two elements forming more than one compound.

All the stoichiometrical laws, which have hitherto been deduced and explained only by the atomic hypothesis, prove to be direct consequences of the purely empirical

definition of the concept of a substance proper or a chemical individual, and the atomic hypothesis is rendered superfluous for the purpose of this deduction.

According to the energetic theory of the elements, these bodies are explained as being distinguishing points of maximum stability relative to all adjacent conceivable substances. If we assume that the two characteristics are sufficient to determine the nature of a substance, and if we put these characteristics in plane co-ordinates horizontally and erect vertical lines in every point of the plane to express the free energy of the corresponding substances, we get a continuous surface like the stalactitic ceiling of a cavern, the end of each stalactite representing an element. A drop of water hanging on the ceiling represents by its flow the possible changes between the elements. To bring the drop from one stalactite to another, it must be raised above the pass between the two stalactites. This requires a certain concentration of free energy, and the practical impossibility of changing one element into another is due to the practical impossibility of bringing about the required concentration of energy. Now, with radium and other unstable elements, energy is not a relative minimum compared with the adjacent possible substances, but only a change in the slope of the surface which represents it. The drop of water can flow of its own accord from the point of the element, and will only lessen its speed on passing through this point. As the drop arrives finally at the end of the stalactite of helium, which is very low indeed (because helium cannot even form compounds), the enormous development of energy connected with this transmutation is explained at once. From the general law, that the most stable point of a changing system is not reached directly, but only through all the possible stages of unstable forms of the system, the intermediate formation of new and transitory substances, and of new and transitory forms of energy is also explained, again without any application of hypothetical atomic concepts.

In presenting the Faraday Medal to Professor Ostwald at the conclusion of the lecture, the PRESIDENT said:—It is now my pleasant duty, Professor Ostwald, in the name of the Society, to offer for your acceptance this medal, which bears the image of Faraday, and which has been struck in commemoration of this occasion.

Prof. DEWAR—You, Sir, have placed upon me the very onerous duty of expressing the grateful thanks of the members of the Chemical Society of London to Professor Ostwald for the magnificent way in which he has discharged the duties of Faraday Lecturer, and the honour he has done us in accepting that office. You, Sir, have pointed to the great galaxy of talent with which he must be associated for all time, in referring to the great names of those who in the past have occupied the position. Now I am quite sure that every chemist in England, when he heard that Professor Ostwald was selected for this office, had a hopeful anticipation that he would rival his compeers. You, fellow-workers, have listened to the mode in which he has passed through this great ordeal, and I am sure that you will all acknowledge the splendid achievement of his success. I think, Sir, we might even go farther and say that no greater enjoyment could have been given to Faraday, if we could imagine his being present here this evening, than to have received Professor Ostwald; for his life and spirit has been the spirit of Faraday, and that has led to his great position as a worker and to the establishment of his great school of physical chemistry. We may rest assured that the man who has done so much to develop the electro-chemical theory and the theory of dielectrics which originated in this place would have been a delight to Faraday. But I think we ought to go farther, and say that, while in the past we have had the eulogy of Faraday, while we have had admirable summations of his character and work by his admirers, we have never before had such an original pronouncement. We have received for the first time an original contribution that is likely to give us matter for thought for many a day to come. I think that

the selection of the subject of the address in itself would have delighted Faraday, who again and again returned to the discussion of the nature of the elementary bodies. I was curious to ascertain what was the last pronouncement which Faraday gave in this room with regard to the elements; and I found it in a Friday evening lecture in the year 1836 (subsequent to his discovery of definite electrolysis), entitled "The Nature of the Chemical Elements." The last sentence is most interesting. He says:—"Thus, either present elements are the true elements, or else there is the probability before us of obtaining *some more higher and general power* of Nature even than electricity, which at the same time might reveal to us an entirely *new* grade of the elements of matter now hidden from our view and almost from our suspicion." That was Faraday's view sixty-eight years ago, given in this very room and at that very table. It is quite clear that at that time he would have been staggered if he could have known that a substance with which he was familiar, in the form of an oxide, and which had been taken for an element—namely, the substance uranium—contained within itself that very spontaneous change into the "new grade of the elements" which Professor Ostwald has so admirably illustrated.

The Professor's stalactite cavern is a cavern of great mystery, and is indeed one in which we may require a great deal more light before we can get acclimatised to the surroundings. But that does not withdraw one iota from the originality and the brilliancy of the lecture which we have had to-night, and I ask you, Sir, to allow me, on behalf of the Chemical Society and the world of English science, to propose a vote of thanks to Professor Ostwald for the noble way in which he has discharged his duty.

DR. THORPE—Ladies and Gentlemen, I rise at the bidding of the President to discharge what I feel to be a very honourable duty, namely, to join with Professor Dewar in giving expression to the gratitude which the Fellows of the Chemical Society feel to Professor Ostwald for the Faraday Lecture which he has given to-night. The President has told you that one characteristic of the Faraday Lectures is that they are given in this hall, a hall which, of course, is hallowed by the associations of Faraday. Now, if anybody will cast a retrospective glance over the Faraday Lectures, they will find that there is one fundamental conception common to them all. However different they may seem at first sight, they are all concerned fundamentally with the idea of the essential nature of chemical action and the essential nature of elements and compounds. Now, if those two things be the characteristics, namely, that the Faraday Lectures are given here, and that they are all concerned with that fundamental idea—Professor Dewar would seem to have proved that Faraday himself was the first Faraday Lecturer. I venture, however, to point out that in reality the first Faraday Lecturer was John Dalton, for it was from that very place which Professor Ostwald has just occupied that John Dalton, in the winter of 1809, developed before such an audience as we have here to-night the conceptions which are immortally associated with his name. It was to an audience which, I suppose, may have comprised men like Young, Wollaston, Sir Joseph Banks, and Humphry Davy himself, that John Dalton, in his characteristic, simple, straightforward, lucid way, laid before a Royal Institution audience the first "Faraday Lecture." I have the greatest possible pleasure, Ladies and Gentlemen, in echoing the sentiment which has been so eloquently expressed by my colleague, Professor Dewar, in asking you to tender to Professor Ostwald our most grateful thanks for the intellectual treat which he has afforded us to-night.

LORD RAYLEIGH—I wish the President had seen fit to call upon someone more conversant than I am with the course of chemical thought and speculation to speak to this resolution. I feel that I follow only at a distance, and, although anyone who has heard Professor Ostwald to-night could not fail to take up some valuable ideas, I think most of us must feel that a good deal of thought is required before

we should be in a position to do justice to what has been laid before us. We certainly live in a very interesting time. Twenty or thirty years ago an idea was not uncommon, I think, that we understood tolerably well the general course of Nature, and that all that was wanted was to fill up certain details where difficulties of one sort or another had interfered with our acquiring adequate knowledge. I believe that the feeling at the present day among scientific men, and, perhaps, especially among those scientific men who have taken the largest part in recent work, would be a very much more modest one, and that most of us are quite prepared to recognise that we must face possible revolutions in those ideas which in many cases we have hitherto regarded as most firmly established. It is in connection with such thoughts that I think we recognise the value of what we have heard to-night, and I, certainly, for one shall look forward to studying in detail what has been set before us. I desire most heartily to support the resolution which has been moved and seconded.

The motion was then put by the PRESIDENT and carried with acclamation.

PROF. OSTWALD—Mr. President, Ladies, and Gentlemen, now that my task of reading from print is at an end, I feel a difficulty in going some steps farther into unknown English periods. I cannot bring sufficient English together to express my deep feeling of thanks for the honour which has been shown to me in the way in which you have received my ideas. I feel that I was not mistaken when I tried to bring this work into your own land in the face of one of the best things ever done in science, I mean the atomic hypothesis. My purpose was to show that the atomic hypothesis is no longer necessary to explain the stoichiometrical laws. But the views arrived at, and the progress attained by the use of the atomic hypothesis, cannot be destroyed. They have directed the discoveries of chemistry for a whole century, and if any hypothesis can do good, this one has done so. Therefore I do not think this hypothesis need be interred yet, and when the time comes it should be interred with the greatest honour.

#### Ordinary Meeting, Wednesday, April 20th, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MESSRS. G. E. P. Broderick, H. W. Gadd, and T. S. Price were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Albert Ernest Bellars, B.A., Magdalene College, Cambridge; Herbert Frank Brand, M.A., B.Sc., 13, Clifton Road, Brockley, S.E.; Thomas Alfred Gerard, 122, Foxhall Road, Nottingham; James Gray Gilchrist, M.A., 48, Ovington Street, Chelsea, S.W.; John Haslam Johnston, M.Sc., Public Offices, Hampton, Middlesex; Alfred Pell, 44, Cumballa Hill, Bombay; David John Pinkerton, 17, Orbiston Street, Motherwell; Percy Richard Sanders, West Cliffe, Seaford, Sussex; James Alfred Wilkinson, M.A., Transvaal Technical Institute, Johannesburg.

THE PRESIDENT stated that the Council had resolved to present the following Address to Sir Henry E. Roscoe on the occasion of the celebration of the Jubilee of his Doctorate, on Friday, April 22nd, 1904, fifty years having elapsed since he graduated as Doctor of Philosophy at Heidelberg on March 15th, 1854.

TO SIR HENRY ENFIELD ROSCOE, Ph.D., LL.D., D.Sc.,  
F.R.S.

The Officers and Council of the Chemical Society desire to join the rest of your Scientific friends in offering hearty congratulations on the attainment of the Jubilee of your Doctorate.

More than forty years ago, when there were few schools of scientific chemistry in this Country, by your teaching

and example you not only revived the fortunes of the College in Manchester to which, during so many years, you were attached, but assisted in arousing a new spirit in the other teaching institutions of Great Britain.

They recall with pleasure and gratitude the services you rendered to the Society when, many years later, you became its President, and by your genial personal influence assisted so materially in promoting its activity and usefulness.

They wish you many years of health to enjoy the further developments of Scientific Research and the further applications of Scientific Knowledge to useful purposes, to which the greater part of your life has been devoted.

(Signed). WILLIAM A. TILDEN, *President*.  
ALEXANDER SCOTT, *Treasurer*.  
W. PALMER WYNN } *Secretaries*.  
M. O. FORSTER }  
WILLIAM RAMSAY, *Foreign Secretary*.

Of the following papers, those marked \* were read.

\*58. "The Vapour Density of Hydrazine Hydrate." By ALEXANDER SCOTT.

The determinations by Curtius and Schulz (*Journ. Prakt. Chem.*, 1890, [2], xlii., 529) of the vapour density of hydrazine hydrate at various temperatures are interpreted by them to indicate something quite anomalous in the behaviour of this substance in the state of vapour. They state that at 100° the vapour density determined by Hofmann's method corresponds with the molecule  $N_2H_4 \cdot H_2O$ , and at 170° at atmospheric pressure to  $N_2H_4 + H_2O$ , but that at higher temperatures larger molecules are largely regenerated, and that at very high temperatures the vapour density indicates a molecular weight double that of the original. The author finds that at 98.8° the vapour density is 15.8 instead of 25 as required by  $N_2H_6O$ , at 138° the dissociation into  $N_2H_4 \cdot H_2O$  is complete, and that at higher temperatures a certain amount of decomposition into nitrogen, ammonia, and water occurs. If the vapour densities by V. Meyer's method were determined in an atmosphere of air, oxidation of the hydrazine would ensue, leading to completely erroneous results, even at comparatively low temperatures, but by using nitrogen this source of error is avoided.

\*59. "The Combining Volumes of Carbon Monoxide and Oxygen." By ALEXANDER SCOTT.

The ratio by volume in which carbon monoxide combines with oxygen has been determined by means of the same apparatus as was employed by the author in estimating the composition by volume of water (*Phil. Trans.*, 1893, clxxxiv., A, 543). The results of the author's experiments indicate that the molecular concentration of carbon monoxide is slightly greater than that of oxygen, the combining volumes being  $CO : O :: 1.9985 : 1$  with carbon monoxide from calcium oxalate, and  $1.9994 : 1$  with that from formic acid. Applying a correction for this to Lord Rayleigh's recently published value for the density of carbon monoxide so as to obtain its molecular weight, we obtain  $CO = 27.99$  and  $C = 11.99$ .

\*60. "A Revision of the Atomic Weight of Rubidium." By EBENEZER HENRY ARCHIBALD.

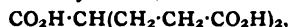
Commercial rubidium iodide was converted into the dichloride ( $RbCl_2$ ) and fractionally crystallised many times until the salt had become spectroscopically free from potassium, when the product was divided into four portions, which each received a different number of additional fractionations. In order to remove the caesium, the rubidium was either repeatedly precipitated as chloride with hydrogen chloride or the hydrogen tartrate was fractionally crystallised. After being fused and bottled by means of Richards's bottling apparatus (*Proc. Am. Acad.*, 1896, xxxii., 55), four samples of rubidium chloride purified by these processes were analysed by precipitating the chlorine with silver nitrate, estimating the amount of silver required for complete precipitation, and also the amount of silver chloride produced. The mean values of the atomic weight of rubidium obtained from 14 analyses were 85.490 and

and 85.484 from the ratios  $AgCl : RbCl$  and  $Ag : RbCl$  respectively ( $O = 16.0$ ).

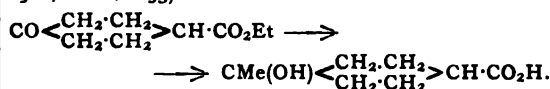
Analysis of rubidium bromide led to the value 85.483, obtained from either of the ratios  $AgBr : RbBr$  or  $Ag : RbBr$ .

\*61. "Experiments on the Synthesis of the Terpenes. Part I. Synthesis of Inactive Terpeneol, Dipentene, and Terpin Hydrate." By WILLIAM HENRY PERKIN, jun.

It was recently shown (*Trans.*, 1904, lxxxv., 416) that pentane- $\alpha\gamma$ -tricarboxylic acid,—

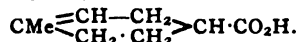


when digested with acetic anhydride and subsequently distilled, is converted into  $\delta$ -ketohehexahydrobenzoic acid. The ester of this acid reacts readily with magnesium methyl iodide, yielding, among other products, *cis*- $\delta$ -hydroxyhexahydro-*p*-toluic acid (compare Stephan and Helle, *Ber.*, 1902, xxxv., 2153):—

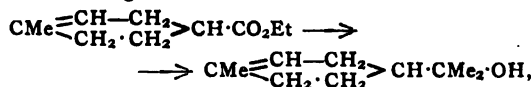


This acid melts at 153°, and on distillation is readily converted into its lactone, a solid, crystalline substance, which melts at 70° and distils at about 185° under 150 m.m. pressure.

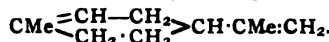
When the hydroxy-acid (or its lactone) is left in contact with fuming aqueous hydrobromic acid it is converted into  $\delta$ -bromohehexahydro-*p*-toluic acid, a crystalline substance which melts at 126°, and which, when digested with pyridine or with sodium carbonate, yields  $\Delta^1$ -tetrahydro-*p*-toluic acid (m. p. 99°):—



If the ester of this acid is mixed with an excess of an ethereal solution of magnesium methyl iodide, and the product, after remaining for twenty-four hours, is treated with dilute hydrochloric acid, a colourless oil is obtained, which distils at 133° under 60 m.m. pressure, and has a pronounced odour of lilac. That the above reaction takes place according to the scheme:—



and that the oil obtained is therefore inactive *terpeneol* is clearly proved by the following facts. It yielded, on analysis, numbers agreeing with the formula  $C_{10}H_{18}O$ , and when digested with potassium hydrogen sulphate, was converted almost quantitatively into dipentene,—



The *dipentene* thus synthesised was found to be identical in all respects with that obtained from ordinary *terpeneol* by the same process. It distilled at 181—182°, had a characteristic odour of lemons, and yielded with bromine, *dipentenetetra*bromide,  $C_{10}H_{16}Br_4$  (m. p. 125°), and, with hydrogen chloride, *dipentene dihydrochloride*,  $C_{10}H_{16} \cdot 2HCl$  (m. p. 48—50°).

Furthermore, the synthetical *terpeneol* was slowly converted by treatment with dilute sulphuric acid into *terpin hydrate*,  $C_{10}H_{18}(OH)_2 \cdot H_2O$  (m. p. 118°), from which *terpin* itself,—



was readily obtained by dehydration.

These experiments are being continued and extended to the study of the behaviour of ethyl  $\gamma$ -ketohehexahydrobenzoate and other similarly constituted substances towards magnesium methyl iodide.

\*62. "Lavorotatory Modification of Quercitol." By FREDERICK BELDING POWER and FRANK TUTIN.

Quercitol,  $C_6H_{12}O_5$ , has hitherto only been found in the

fruits (acorns) of certain species of *Quercus*, in which it exists as a dextrorotatory modification. The laevorotatory modification described by the authors was obtained from the leaves of *Gymnema sylvestre* (Br.), a plant belonging to the family of *Asclepiadaceae*, and indigenous to Banda and the Deccan Peninsula.

*l*-Quercitol is a colourless, crystalline substance, which, when crystallised from water, has the formula  $C_6H_{12}O_5 \cdot H_2O$ ; it loses its molecule of water at  $110^\circ$ , melts at  $174^\circ$ , and has  $[\alpha]_D -73.9^\circ$ . The dried substance separates from ethyl alcohol in the anhydrous state. *Pentaacetyl-l-quercitol*,  $C_6H_7(O \cdot C_2H_5O)_5$ , crystallises from dilute alcohol in needles, melts at  $124-125^\circ$ , and has  $[\alpha]_D -26.0^\circ$ . *Pentabenzoyl-l-quercitol*,  $C_6H_7(O \cdot C_7H_5O)_5$ , separates from a mixture of alcohol, ethyl acetate, and petroleum in needles containing a molecule of alcohol, which, when heated slowly, melt at  $148^\circ$ ; it has  $[\alpha]_D -79.0^\circ$ . On oxidising *l*-quercitol with sodium hypobromite and treating the product with phenylhydrazine, *diketotrihydroxyhexahydrobenzenedihydroxone*,  $C_6H_5(OH)_3 \cdot (N \cdot NH \cdot C_6H_5)_2$ , was obtained, which separates from alcohol in yellow needles and melts at  $209^\circ$ . On oxidising with cold potassium permanganate solution, malonic acid was obtained, and was identified by means of its ethyl ester.

Kiliani and Schaefer (*Ber.*, 1896, xxix, 1762) have shown that *d*-quercitol is a pentahydroxyhexahydrobenzene, by the formation, on the one hand, of malonic acid, and, on the other, of a diketone,  $C_6H_8O_5$ . The substance isolated by the authors, designated as *l*-quercitol, is, therefore, one of the eight possible isomerides of pentahydroxyhexahydrobenzene, but, until a further number of these have been isolated, it will be impossible to assign to either of the known quercitols a definite configuration.

\*63. "The Constituents of the Essential Oil of Californian Laurel." By FREDERICK BELDING POWER and FREDERICK HERBERT LEES.

The Californian laurel, *Umbellularia Californica* (Nuttall), a handsome evergreen tree, occurring in California and Oregon, is also known as "mountain laurel," "cajeput," "spice tree," "California olive," "California bay tree," and "pepper-wood."

The essential oil of the leaves had a pale yellow colour and an odour which was at first agreeably aromatic, but when more strongly inhaled became exceedingly irritating to the mucous membranes of the nose and eyes, producing the effects that have previously been described. The oil had a sp. gr.  $0.9483$  at  $16^\circ/16^\circ$ , and  $[\alpha]_D -22^\circ$  in a 100 m.m. tube; it was completely soluble in 1.5 parts of 70 per cent alcohol. It was found to contain an inappreciable amount of esters and a very small quantity of a mixture of free fatty acids, including formic acid. The essential constituents of the oil were found to be: eugenol, *l*-pinene, cineol, saffrole, eugenol methyl ether, veratric acid, and a new, unsaturated, cyclic ketone, *umbellulone*,  $C_{10}H_{14}O$ , which is a colourless liquid with a somewhat mint-like odour, having in a high degree the peculiar pungency of the original oil. It boils at  $219-220^\circ$  (corr.), has a sp. gr. of  $0.9581$  at  $15/15^\circ$ , and  $[\alpha]_D -37^\circ$ . This ketone does not react normally with semicarbazide and hydroxylamine, affording with the former, *semicarbasidodihydro-umbellulonesemicarbazone*,



(m.p.  $217^\circ$ ), and with the latter, *hydroxylaminodihydro-umbelluloneoxime*,  $C_{10}H_{15} \cdot (NOH) \cdot NH \cdot OH$  (compare *Ber.*, 1897, xxx., 230, 251; 1900, xxxiii., 562; and 1903, xxxvi., 4377).

The relative proportions of these constituents of the oil were approximately as follows:—Umbellulone, 60; cineol, 20; eugenol methyl ether, 10; pinene, 6, and eugenol, 1.7 per cent respectively, together with a very small amount of saffrole.

\*64. "Some Derivatives of Umbellulone." By FREDERICK HERBERT LEES.

With the view of elucidating the constitution of um-

bellulone,  $C_{10}H_{14}O$ , the new ketone isolated by Power and the author from the essential oil of Californian laurel, the following derivatives have been prepared.

Umbellulone combines directly in the cold with only 2 atomic proportions of bromine, forming *umbellulone dibromide*,  $C_{10}H_{14}OBr_2$ , an unstable oil. Umbellulone would therefore appear to contain only one ethylenic linking, and in consideration of this circumstance and the fact that, like the  $\alpha\beta$ -unsaturated ketones generally, it behaves abnormally towards hydroxylamine and semicarbazide, one molecule of the ketone interacting with two molecules of these bases respectively (compare preceding abstract), it is regarded as an  $\alpha\beta$ -unsaturated cyclic ketone containing two closed rings.

When umbellulone dibromide is heated under diminished pressure, it loses hydrogen bromide, yielding an *unsaturated bromo-ketone*,  $C_{10}H_{13}OBr$  (b. p.  $140-145^\circ/20$  m.m.), and *dibromodihydroumbellulone*,  $C_{10}H_{14}OBr_2$ , which forms needles melting at  $119-119.5^\circ$  and having  $[\alpha]_D +6.4^\circ$  in chloroform. When the unsaturated bromo-ketone is reduced with zinc dust and acetic acid, a *saturated ketone*  $C_{10}H_{16}O$  (b. p.  $214-217^\circ$ ) is produced; the *semicarbazone* of the latter,  $C_{10}H_{16}N \cdot NH \cdot CO \cdot NH_2$ , forms needles which melt at  $171-172^\circ$ .

When dibromodihydroumbellulone is reduced with zinc dust and acetic acid, it is converted into *bromodihydro-umbellulone*,  $C_{10}H_{15}OBr$ , which forms needles melting at  $58-59^\circ$  and having  $[\alpha]_D +70.1^\circ$  in chloroform. When this bromo-derivative is reduced with sodium and alcohol, it gives *tetrahydroumbellulol*,  $C_{10}H_{19}OH$  (b. p.  $207-208^\circ/760$  m.m.), which has a sp. gr.  $0.9071$  at  $15/15^\circ$  and  $[\alpha]_D -6.6^\circ$ .

Umbellulone is easily oxidised by cold potassium permanganate, yielding a *lactone*,  $C_9H_{12}O_2$ , together with several acids which have not yet been investigated.

The author wishes to reserve the further investigation of umbellulone.

(To be continued).

Conditions of Formation and the Solubility of Cuprosodic Sulphate.—J. Koppel.—Nassol and Maldés (*Comptes Rendus*, vol. cxxxiii., p. 287), in their researches on the solubility of mixtures of sulphate of copper and sulphate of soda, observed that at a low temperature the composition of a solution doubly saturated with regard to the two salts is independent of the ratio of the masses of the two salts present; but that as soon as the temperature is sufficient for the anhydrous modification of sulphate of soda to take rise (from  $23^\circ$  to  $32^\circ$ , according to the mixture), the composition of the solution varies with the relative proportions of the two salts used. Now, the phase rule teaches us that such a result can only be observed in the case where the two salts give place to either isomorphous mixtures or to a double salt. As there is no question of isomorphism between  $SO_4Cu_5H_2O$ , which is triclinic,  $SO_4Na_2 \cdot 10H_2O$ , which is clinorhombic, and  $SO_4Na_2$ , which is orthorhombic, the observations of Nassol and Maldés point to the existence of a double salt. This double salt being apparently formed, with a loss of water, from two hydrated salts, it follows from a law given by van't Hoff (*Bull. Soc. Chim.*, vol. xxviii., p. 218) that its production should be brought about by a rise of temperature. The author has succeeded in isolating this double salt, which corresponds to the formula  $SO_4Na_2 \cdot SO_4Cu_2 \cdot 2H_2O$ ; the thermometric and dilatometric examination of its formation and of its decomposition shows that its transformation point is  $16.7^\circ$ . This salt is identical with a natural mineral, *kr hnkite*. The author has determined its solubility, both in pure water and in solutions of one or the other of the two component salts. The results obtained below the transformation point agree with those obtained by Nassol and Maldés; those obtained above this point are decidedly different, these chemists having operated on mixtures in course of transformation without waiting until equilibrium had been reached.—*Zeit. Physik. Chem.*, vol. xlii., p. 1.

# NOTICES OF BOOKS

*Entropy.* By JAMES SWINBURNE. London: A. Constable and Co., Limited. Pp. 137.

In his preface the author states that his reason for writing this book is that it is very much wanted, as there is no work in English, so far as he is aware, that gives a correct definition of entropy. In his first chapter he says:—"Most writers, after defining entropy incorrectly, unconsciously depart from their definitions in dealing with the  $\theta\phi$  diagram, so that the two errors approximately cancel out." This possibly accounts for our own unfortunate condition, in which, however, we happen to know that we are not unique, for before reading this book we were quite ignorant of what entropy is; nor, to be candid, are we quite certain that we are much wiser now.

On page 8 we read:—"Entropy may be defined thus—Increase of entropy is a quantity which, when multiplied by the lowest available temperature, gives the incurred waste."

Strictly speaking, this is not a definition of entropy; but if we interpret the statement correctly, it supplies data by which a definite idea of the nature of entropy may be obtained, and also supplies the means by which its exact quantity with certain restrictions may be calculated in terms of known physical units.

On page 105 we find the following statement:—"It may seem strange that a standard state has to be taken for zero entropy instead of simply finding the total entropy of a body and stating it. The usual reason given is that the total entropy of a body is unknown. The real reason is that the entropy of a body is necessarily infinite. If a body of unit mass is imagined at zero temperature, that is taken as zero entropy, because as no heat can be given out no further reduction of entropy is possible, to heat it up to any finite temperature  $\theta$ , the increase of entropy necessary is—

$$\int_0^{\theta} \frac{d\theta}{\theta} = \phi,$$

$$\phi = \log \theta_1 - \log \theta_0;$$

but  $\log \theta_0$  or  $\log 0$  is negative infinity, so the entropy needed to raise the body to any finite temperature is infinite."

This is a surprising statement, and one which we find hard to reconcile with the so-called definition of entropy on page 8. For consider the equation—

$$\int \frac{d\theta}{\theta} = \phi;$$

if this means anything, it means—

$$\theta d\phi = d\theta,$$

and put into ordinary language it reads:—"Increase of entropy multiplied by the temperature equals the increase of temperature; whence it apparently follows that "increase of temperature" is "incurred waste," but it seems safer to conclude that the author's two statements are inconsistent. Moreover, we fail to equate either of them with the statement made by M. Berthelot:—"For example, entropy being represented by  $\int \frac{dQ}{T}$ , one has  $dQ = c dT$ ;  $c$  being the specific heat, which is itself a function of  $T$ ."

M. Berthelot, writing in French, escapes the sweeping condemnation of English works referred to above. If his statement be correct, it follows that the author's are wrong, unless it be conceded that—

$$\text{"Incurred waste"} = dQ.$$

To return to page 105. We see no reason to doubt the usual reason given for not stating the total entropy of a body; it is at present unknown, because the physical constants have not been determined for the whole scale of temperatures. As for the statement that the entropy is

infinite, we see no justification; it may be true; if so, it is surprising.

When we come to the use made of the  $\theta\phi$  (or entropy) diagram, the author writes on more conventional lines, and as he justly remarks, more use might be made of this method of localising and checking waste of heat. The repetition of the diagrams from page to page, so long as the discussion on them lasts, is very convenient.

*The Old Riddle and the Newest Answer.* By JOHN GERARD, S.J., F.L.S. London, New York, and Bombay: Longmans, Green, and Co. 1904.

IN the preface to this volume it is stated that it aims at investigating the assumption on the part of Science that she has solved the riddle of Nature. The first part examines the truth of the law of evolution, using the term in its widest sense, and criticises Prof. Haeckel's "Riddle of the Universe." Here our author shows himself, it must be admitted, skilful in argument and judicious in the conclusions he draws, and undoubtedly there are to be found scientific men who will, in part at any rate, agree with his deductions, though they might not altogether approve of his methods. In the second part he deals with the principle of evolution as applied to organic nature only and with the Darwinian theory of natural selection. But he does not seem to realise that the fact that Darwin made some mistakes does not disprove his whole theory, and isolated quotations from various authors' works as given in this book cannot be taken in any sense as authoritative. And a serious defect in our author's method appears to be the way in which he would lead an uninformed reader to infer that Darwin himself had not realised the weakness in the demonstrative proof of his theory, and explained this weakness in detail in the chapter in the "Origin of Species" on the "Imperfection of the Geological Record." Whether Darwin's answer to these objections, which he himself actually suggested in many instances, is satisfactory, is a question for individual decision, but the matter will take a different aspect in the minds of many if they first read the chapter referred to in the "Origin of Species."

At any rate it may be affirmed that on the whole the author displays an earnest spirit of enquiry and no little skill in the use of the material he has collected.

*Die Elektrolytische Raffination des Kupfers.* ("The Electrolytic Refining of Copper"). By TITUS ULKE, M.E. Translated into German by VIKTOR ENGELHARDT. Halle-a-S.: Wilhelm Knapp. 1904.

THE author of this monograph has had many opportunities of personally visiting some of the most important works in which copper ores are treated by electrolytic means, and has thus made himself acquainted with much interesting and useful information concerning output and arrangement of plant. He has also collected accurate information with regard to so-called secret methods, and has carefully abstracted French, German, English, and American patent literature. Thus the book, which contains several valuable plans, will be found very useful by works managers and others.

The translator has been confronted with a difficulty in performing his task as regards the conversion of the various numbers given into the metric system. After considering the various courses open to him, he has decided to retain the numbers as given in the text, and adds in brackets the values in round numbers in the metric system.

*Classification des Corps Simples.* ("The Classification of Elementary Substances"). By M. HENRI MOISSAN. Paris: Masson et Cie. 1904.

IN this extract from the "Traité de Chimie Minérale," after discussing shortly the definition and nature of an elementary substance, the author proceeds to give a brief historical

account of the theories which have been put forward regarding the classification of the elements from the earliest times up to the present day. The various systems of Thénard, Berzelius, Frémy, Naquet, Mendeleeff, and Lothar Meyer are then explained, and the author's suggestions put forward for grouping the elements into natural families, taking into account their physical and chemical properties. The differences between his and other systems of classification are clearly explained, while the difficulties of a satisfactory solution of the problem are fully recognised.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 14, April 5, 1904.

This issue contains no chemical matter.

*Bulletin de la Société Chimique de Paris.*

Series 3, Vol. xxix., No. 13.

Alloys of Copper and Magnesium.—O. Boudouard.—Already noticed.

The Electrolytic Estimation of Small Quantities of Silver in the presence of a large Amount of Lead.—MM. Arth and Nicolas.—Already inserted in full.

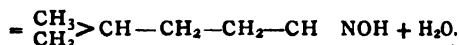
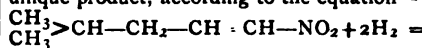
The Volumetric Estimation of the Alkaline Nitroprussiates and of the Soluble Salts of Cadmium.—MM. Fonze-Diacon and Carquet.—Already inserted.

The Toxicity of Nitroprussiate of Soda.—MM. Fonze-Diacon and Carquet.—A mean dose of 0.25 grm. of nitroprussiate of soda, per kilogram. of the animal, will kill a rabbit, either when administered by the mouth or by hypodermic injection. In this way it may be calculated that for an ordinary man of 150 lbs. weight, 17 or 18 grms. of nitroprussiate of soda would be fatal. A post mortem examination shows the presence of hydrocyanic acid in the stomach. It is probable that the nitroprussiate of soda in the presence of the lactic acid in the stomach is decomposed by the gastric glands, as is chloride of sodium; thus an unstable body is formed which would give rise to the formation of prussic acid.

The Degree of Accuracy attained in the Detection of Traces of Arsenic in Organic Matters.—Armand Gautier.—A reply to criticisms of his method. The author points out that he can recover arsenic integrally, even when dealing with such minute quantities as one or two thousandths of a m.grm. in 50 million times its weight of organic matter.

A Method for the Gradual Synthesis of the Fatty Aldehydes.—MM. L. Bouveault and A. Wahl.—An equimolecular mixture of nitromethane and valeral is placed in a flask, and 1 molecule of 30 per cent aqueous caustic potash added a little at a time. The temperature must be kept down to 30°. When all the potash has been added the solution becomes yellow, and is then poured into an excess of dilute hydrochloric acid; an oil is precipitated, which is extracted with ether and distilled. The nitrated alcohol is a pale liquid, boiling at 110° under 15 m.m., but it is not necessary to distil *in vacuo* before transforming in nitroisohexylene, the raw product being sufficiently pure to be treated at once. To effect the dehydration, the product is dissolved in five times its weight of glacial acetic acid in which a quantity of fused chloride of zinc has been already dissolved, equal to half the weight of the nitrated alcohol to be treated; then boil with a vertical condenser for five or six hours. The liquid is then distilled with steam, and the portion floating on the distillate is collected

with ether and distilled *in vacuo*; it consists of nitroisohexylene. It can be reduced by aluminium amalgam or zinc powder and acetic acid, with the formation of isobutyl-acetaldoxime. Thus the reduction of the nitrated derivative of the non-saturated carbide has given the oxime as a unique product, according to the equation—



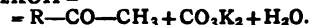
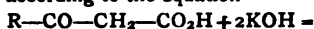
A Series of New Acids with an Acetylenic Function.—Ch. Moureu and R. Delange.—Already noticed.

The Hydration of the Acetylenic Acids. Synthesis of Caprylic and Pelargonic Acids.—Ch. Moreu and R. Delange.—The acetylenic acids,  $\text{R}-\text{C}\equiv\text{C}-\text{CO}_2\text{H}$ , are capable of fixing four atoms of hydrogen by the opening of the triple bond, giving the corresponding saturated acids,  $\text{R}-\text{CH}_2-\text{CH}_2-\text{CO}_2\text{H}$ . This has been specially done with the two natural fatty acids: caprylic acid,  $\text{CH}_3-(\text{CH}_2)_6-\text{CO}_2\text{H}$ , which exists in the form of a glyceride in certain natural fatty bodies, particularly in butter, by starting with amylpropionic acid,  $\text{CH}_3(\text{CH}_2)_4-\text{C}\equiv\text{C}-\text{CO}_2\text{H}$ ; and with pelargonic acid,  $\text{CH}_3-(\text{CH}_2)_7-\text{CO}_2\text{H}$ , starting with hexylpropionic acid,  $\text{CH}_3-(\text{CH}_2)_5-\text{C}\equiv\text{C}-\text{CO}_2\text{H}$ .

A New Fatty Acid,  $\gamma, \gamma', \gamma''$ -Trimethylbutyric Acid.—Ch. Moureu and R. Delange.—Dissolve 14.20 grms. (1 molecule) of trimethyltetrollic acid in 1000 grms. of absolute alcohol, and gradually add 100 grms. of sodium to the boiling solution. Heat with a vertical condenser until all the sodium has reacted. Distil off the excess of alcohol, completing the operation *in vacuo*, and throw the dry residue, a little at a time, into 1000 c.c. of water. The alcohol formed by the decomposition of the alkaline ethylate is separated by distillation, and the aqueous residue cooled. This is carefully treated with a slight excess of dilute sulphuric acid, causing an oily appearance. The mixture is exhausted with ether, and the ethereal solution washed first with a concentrated aqueous solution of chloride of calcium, then with water; dry over anhydrous sulphate of soda, evaporate the ether, and rectify the residue at atmospheric pressure. This new acid congeals almost instantly in a freezing mixture, and by spontaneous heating it melts between  $-1^\circ$  and  $+3^\circ$ . Its density at  $20^\circ$  is 0.9129, and at  $0^\circ$  in the supercooled state 0.9238.

The Hydration of Acids with an Acetylenic Function. New Method for the Synthesis of the Non-substituted  $\beta$ -Ketonic Acids and Ethers.—Ch. Moureu and R. Delange.—Already noticed.

The Decomposition of the Acetylenic Acids by the Alkalis.—Ch. Moureu and R. Delange.—When we prepare the non-substituted  $\beta$ -ketonic acids,  $\text{R}-\text{CO}-\text{CH}_2-\text{CO}_2\text{H}$ , by heating the acetylenic acids,  $\text{R}-\text{C}\equiv\text{C}-\text{CO}_2\text{H}$ , with a caustic alkali in alcoholic solution, we always observe the production of a certain quantity of the corresponding acetone,  $\text{R}-\text{CO}-\text{CH}_3$ . This is formed by the normal decomposition of the  $\beta$ -ketonic acid, which is formed according to the equation—



This acetone is found in solution in the ether with which we shake the alkaline liquor, resulting from the addition of an excess of water to the alcoholic mixture, as soon as the reaction is terminated. It is isolated by distillation. The author then goes on to describe in detail the method of treating several acetylenic acids in this manner, such as propylpropionic, amylpropionic, isopropylpropionic, and other acids.

General Observations on the Acetylenic Acids,  $\text{R}-\text{C}\equiv\text{C}-\text{CO}_2\text{H}$ .—Ch. Moureu and R. Delange.—The authors point out the close relationship between the acetylenic acids and the non-substituted  $\beta$ -ketonic acids.

The Amylchloracrylic Ethers.—Ch. Moureu and R. Delange.—A solution of 10 grms. of amylpropionic acid

in 50 grms. of absolute alcohol is saturated at  $0^{\circ}$  with a current of dry hydrochloric acid gas. After twenty-four hours the mixture is thrown into an excess of water, the whole shaken up with ether, the ethereal layer washed with water, dried over anhydrous sulphate of soda, and distilled. Thus 11.5 grms. of a colourless oil are collected which has the same proportion of chlorine as amylochlorate of ethyl,  $C_5H_{11}-C_2HCl-CO_2C_2H_5$ . The authors show that the chlorine must be in the  $\beta$  position with regard to the carboxethyl in their amylochloracrylic ether.

**The Nitric Ethers of the Acid Alcohols.**—H. Duval. —The author has shown that the analysis of raw nitrate of glycolic acid gives too high a figure for the carbon; as the water was correct, he considered that there must be another product formed richer in carbon, resulting from a condensation of the glycolic acid; he also showed that there was an oil deposited during the crystallisation of the nitrate of glycolic acid. The examination of this oily compound led him to admit the formation of nitrate of acetoxyacetic acid answering to the formula  $CH_2ONO_2-COOCH_2-CO_2H$ , which is the nitrate of the ether obtained by the elimination of a molecule of water between the alcohol and the acid formations of the two molecules of glycolic acid.

**The Action of Hydrated Oxide of Bismuth on the Acid Isomers of Gallic Acid, and its combination with Pyrogallalcarbonic Acid.**—Paul Thibault. —Bismuthopyrogallalcarbonic acid was prepared by reacting in the cold with an excess of pyrogallalcarbonic acid on pure gelatinous oxide of bismuth in the presence of water; after contact for ten days in a sealed tube, it was opened, decanted, and washed with cold water, or better still with alcohol, after making sure that the pyrogallalcarbonic acid is in excess; dry *in vacuo* over sulphuric acid, or in the oven. Analysis shows the formula to be  $C_7H_5-O_6Bi$ . It is a golden yellow powder, crystallised in needle-like prisms, soluble in strong acids and caustic alkalis, insoluble in acetic acid, even when boiling, and in the neutral solvents such as alcohol, ether, chloroform, &c. It decomposes at about  $195-200^{\circ}$ , and its density at  $18^{\circ}$  is 3.51.

**Tetraphenylbutanediol and its Products of Dehydration.**—Armand Valeur. —Already noticed.

**The Action of Sulphur on the Organo-magnesian Compounds.**—Henri Wuyts and G. Cosyns. —The author describes the action of sulphur on ethyl iodide of magnesium, and on phenylbromide of magnesium; he also describes a method for the preparation of thiophenol by the reduction of disulphide of phenyl.

**Influence of its Surroundings on Vegetable Acidity.**—E. Charabot and A. Hebert. —In the young plant the ash of the leaves and stem is more alkaline than that of the roots; as the plant develops the alkalinity decreases in the former and increases in the latter, which finally become the most alkaline of the two. In fact, the mineral salts increase the proportion of combined acids in the aerial portion of the plant.

## MISCELLANEOUS.

**South-Western Polytechnic, Chelsea.**—Earl Cadogan, K.G., presents the Prizes and Certificates to Students of Evening Classes and Day Colleges for Men and Women to-day, at 8 p.m. On Saturday, May 7th, at 7 p.m., a *Conversazione* will be held, including scientific lectures and demonstrations, exhibits in Science, Art, and Domestic Economy, gymnastic displays, and musical entertainments. The laboratories and workshops will be open for inspection. Admission by ticket.

**Forthcoming Books.**—The *Electrician* Printing and Publishing Co., Salisbury Court, London, announce that they will issue early in May an important work by Mr.

F. Soddy, M.A., entitled "Radio-Activity: an Elementary Treatise from the Standpoint of the Disintegration Theory." This is intended as a text-book for students and others interested in the fascinating research work connected with Radium and other radio-active substances. The same Company will issue in a few days a book by Prof. S. Lemström entitled "Electricity Applied to Agriculture and Horticulture." In this work Prof. Lemström describes the results of a number of important experiments in England, France, Finland, and Sweden on the value to the cultivator of the electric current in accelerating the growth, improving the quality, and increasing the yield of farm, orchard, market garden, and glasshouse crops.

## MEETINGS FOR THE WEEK.

- MONDAY, 9th.**—Royal Institution, 5. General Monthly Meeting. Society of Arts, 4.30. (Cantor Lectures). "The Majolica and Glazed Earthenware of Tuscany," by Prof. R. Langton Douglas, M.A.
- TUESDAY, 10th.**—Royal Institution, 5. "Meteorites," by L. Fletcher, F.R.S., &c. Society of Arts, 8. "Crystalline Glasses and their Application to the Decoration of Pottery," by William Burton, F.C.S.
- WEDNESDAY, 11th.**—Society of Arts, 8. "Early Painting in Miniature," by Richard R. Holmes, C.V.O.
- THURSDAY, 12th.**—Royal Institution, 5. "Great Britain and Europe (1763-1793)," by Arthur Hassall, M.A. Society of Arts, 4.30. "British Grown Tea," by A. G. Stanton.
- FRIDAY, 13th.**—Royal Institution, 9. "The Queen Victoria Memorial," by M. H. Spielmann, F.S.A.
- SATURDAY, 14th.**—Royal Institution, 3. "Sonata Style and the Sonata Forms" (with Musical Illustrations), by Donald Francis Tovey, B.A.

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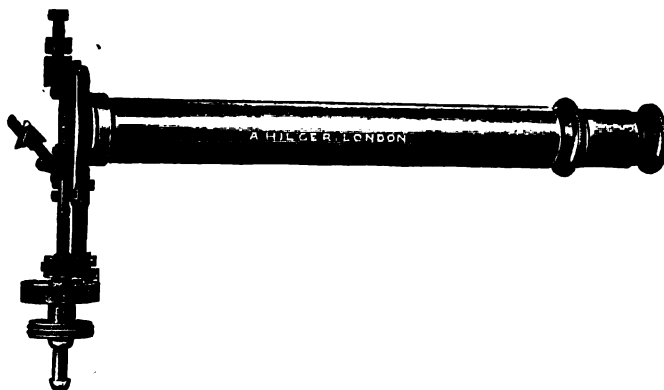
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### CONTENTS.

| ARTICLES:—                                                                                                                                                                                      | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Volumetric Estimation of Cyanogen, by John McDowall ..                                                                                                                                      | 129  |
| The Detection of Chlorides in the presence of Bromides, by Chapman Jones, F.I.C., &c.....                                                                                                       | 129  |
| The Estimation of Organic Matter in Water—Disadvantages of Filtering through Paper before the Analysis, by C. Lenormand Technical Research and the College System, by W. P. Dreyer, F.I.C. .... | 129  |
| The Decomposition of the Final Fractions of Monazite into Components, and the Preparation of Pure Gadolinium Oxide, by R. Marc.....                                                             | 230  |
| PROCEEDINGS OF SOCIETIES—                                                                                                                                                                       |      |
| CHEMICAL SOCIETY .....                                                                                                                                                                          | 235  |
| THE FARADAY SOCIETY .....                                                                                                                                                                       | 236  |
| OBITUARY.—Prof. A. W. Williamson, F.R.S. ....                                                                                                                                                   | 237  |
| NOTICES OF BOOKS .....                                                                                                                                                                          | 237  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                                                                     | 237  |

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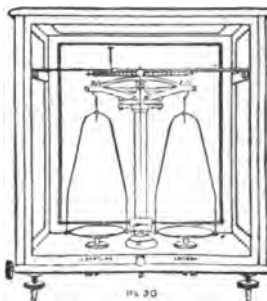
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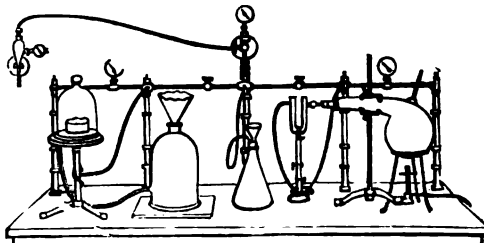
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2320.

## THE VOLUMETRIC ESTIMATION OF CYANOGEN.

By JOHN McDOWALL,  
Chemist to the Backus and Johnston Co., Lima, Peru.

When the blue solution, produced by adding ammonia to a cupric salt, is added to potassium cyanide, the colour disappears until a certain quantity of the coloured solution has been added; then it ceases to be decolourised. On account of the intense colour it seemed that it would make a good standard solution for volumetrically determining the amount of cyanogen present in the presence of chlorides.

The standard solution was made by dissolving 25 grms. of copper sulphate and pouring the solution into a litre flask, then adding distilled water till the flask was half filled; ammonia was then added till a clear blue liquid was produced; the liquid was then diluted up to the mark. The solution was standardised by weighing out 0.5 gm. chemically pure potassium cyanide, dissolving in 100 c.c. water, and adding 5 c.c. of ammonia. The blue solution was then run in with constant stirring until the colour began to disappear slowly, and then drop by drop. The finish is very sharp, one drop being sufficient to tinge the solution a light blue colour, very conspicuous against a white tile.

The following experiments were made to see if chloride had any action. Three beakers were marked respectively I., II., III. Into I. 1 gm. sodium chloride, 2 grms. were added to II., 3 grms. to III.; weighed quantities of cyanide were also added, and 100 c.c. water and 5 c.c. ammonia to each beaker. The result was as follows:—

| No.          | KCN added.<br>Grm. | KCN found.<br>Grm. |
|--------------|--------------------|--------------------|
| I. . . . .   | 1.5                | 1.5                |
| II. . . . .  | 0.98               | 0.96               |
| III. . . . . | 0.64               | 0.64               |

This method is therefore as accurate, as the silver assay and chloride do not interfere. It has the advantage over the silver method of not being acted on by light, and is much cheaper, a matter of some importance where many assays of cyanides have to be done daily. It has also a coloured finish, which is preferable to turbidity in volumetric work. It will be useful in mining districts as a speedy and accurate method for determining the purity of the potassium cyanide used for the extraction of gold.

## THE DETECTION OF CHLORIDES IN THE PRESENCE OF BROMIDES.

By CHAPMAN JONES, F.I.C., F.C.S., &c.

THE only difficulty concerning halogen salts in general qualitative analysis is the detection of a small quantity of a chloride in the presence of a bromide. The methods suggested consist, as a rule, either in eliminating the bromine and then testing for the chlorine, or in precipitating both as silver salts and dissolving out the silver chloride. In the first case the separation is rarely if ever sharp, either a little bromine is left behind or a little chlorine is lost, and it is therefore impossible to say whether or not a little chlorine is present. In the other method there is no loss, and this is a very considerable advantage, but in both the presence and the absence of a little chlorine a positive result is generally obtained, and it remains to discover whether a slight turbidity, produced on re-precipitation from the extract, is due to silver chloride or bromide. With suffi-

cient practice and care this is not impossible when using the reagents that have already been proposed, but a more distinct difference between the two salts is very desirable.

During the last year or two I have used ammonium bicarbonate for this purpose with much satisfaction. A saturated aqueous solution of this salt has the advantage that it is more definite than a dilute solution of ammonia or a solution of any other carbonate of ammonia, and also that it shows a greater difference in its behaviour to the two silver salts. Mr. R. H. Beckett, B.Sc., of this College, has carefully confirmed the following statements, using the precipitated silver salts in as nearly as possible the condition in which they generally occur.

If a cold saturated solution of ammonium bicarbonate is poured over silver chloride on a filter-paper, the extract will give a distinct turbidity on acidification with nitric acid. Silver bromide similarly treated gives no turbidity.

By allowing the silver salts to remain in contact with the reagent for a few minutes with occasional agitation, the chloride will give a greater turbidity on acidification, while the treatment of the bromide may be continued sometimes even for half-an-hour before it begins to give a positive result.

If a turbidity is obtained on acidification of the extract, and it is doubtful whether it is due to silver chloride or bromide, its identity may be established in the following way:—The turbid liquid is divided into two parts; to one is added a slight excess of the bicarbonate solution and to the other an equal quantity of water. If the turbidity is due to silver chloride, the ammonium salt will re-dissolve it in a few seconds. If it is due to the bromide, it will remain unaffected for several minutes, if not an hour or more. The part diluted with water is for comparison with the other, as it is difficult to bear in mind the appearance of a turbid liquid, and to duly allow for its dilution.

It is desirable that the ammonium bicarbonate solution be not old, or it will probably have lost some carbonic acid. A boiling solution of sesquicarbonate of ammonia has been used before for dissolving out silver chloride from the bromide, but in this case some of the bromide is always dissolved.

Royal College of Science, London.

## THE ESTIMATION OF ORGANIC MATTER IN WATER. DISADVANTAGES OF FILTERING THROUGH PAPER BEFORE THE ANALYSIS.

By C. LENORMAND.

In a paper which I have recently published (*Bull. Soc. Chim.*, [3], xxix., 810; *CHEMICAL NEWS*, lxxxix., 219), I described a new method for the estimation of the organic matter in both fresh and salt waters.

I wish now to complete the matter, and to draw particular attention to the very considerable errors which are introduced when the waters are filtered through paper before analysis.

I have made these observations during the course of a large number of analyses of sea-water from oyster-beds. I was struck, in fact, with the very great differences obtained in the estimation of the organic matter in cases where the water was or was not filtered.

I have also tried to find whether similar differences were to be found in the case of fresh waters; and I observed that they did exist, and that they were of the same order.

Sea-water, as I have made quite sure by using the method I have described, contains a variable quantity of organic matter, but this is never so considerable as has been generally imagined, except in special cases. For example, at 4 kilometres from Cape Fréhel, the amount present requires 0.900 m.grm. of oxygen per litre to destroy it; at the mouth of the Rance it requires 1.075

m.grms.; in the harbour of Saint-Malo, 1.250 m.grms.; in the Bay of Cancale, 1.433 m.grms.; in the Gulf of Gascogne, at about 200 kilometres from the shore, 1.450 m.grms. But we no longer find these or similar figures if, as a matter of precaution, we remove the suspended solid matter by means of a filter.

The remark I have just made with regard to sea-water is equally applicable to fresh waters, and further on I shall show what unexpected results are obtained with both salt and fresh waters which have been filtered before analysis.

#### Experiment I.

A Laurent filter of half-a-litre capacity was filled with sea-water, and the latter collected in four flasks of 100 c.c. each. The amounts of organic matter passed from 0.9 m.grm. per litre to 9.725 m.grms. for the first 100 c.c. collected, to 4.925 m.grms. for the second, 2.075 m.grms. for the third, and 1.450 m.grms. for the last.

#### Experiment II.

Filters of different makes were filled with water, exactly 100 c.c. of water passed through each, and the organic matter estimated by the same method.

**Sea-water.**—Results obtained with a Water containing 1.150 m.grms. of Organic Matter per Litre.

| Nature of the filter-paper. | Diameter. |         | Ash. | Organic matter. | Original organic matter. |
|-----------------------------|-----------|---------|------|-----------------|--------------------------|
|                             | C.m.      | M.grms. |      |                 |                          |
| Dreverhoff, No. 400 ..      | 15        | 0.2     | —    | 2.200           | 1.150                    |
| Schleicher, hard ..         | 9         | 0.77    | —    | 3.450           | 1.150                    |
| Dreverhoff, No. 311 ..      | 15        | 5.0     | —    | 3.675           | 1.150                    |
| Berzelius, No. 2 ..         | 15        | 3.7     | —    | 4.400           | 1.150                    |
| Dreverhoff, No. 331 ..      | 15        | —       | —    | 5.200           | 1.150                    |
| Dreverhoff, No. 481 ..      | 15        | 8.0     | —    | 6.000           | 1.150                    |
| Laurent .. .. .             | 15        | —       | —    | 7.550           | 1.150                    |

**Fresh Water.**—Results obtained with a Water containing 1.7 m.grms. of Organic Matter per Litre.

| Nature of the filter-paper. | Diameter. |         | Ash. | Organic matter. | Original organic matter. |
|-----------------------------|-----------|---------|------|-----------------|--------------------------|
|                             | C.m.      | M.grms. |      |                 |                          |
| Schleicher, hard ..         | 9         | 0.77    | —    | 2.700           | 1.700                    |
| Dreverhoff, No. 400 ..      | 15        | 0.2     | —    | 2.725           | 1.700                    |
| Berzelius, No. 2 ..         | 15        | 3.7     | —    | 3.925           | 1.700                    |
| Dreverhoff, No. 481 ..      | 15        | 8.0     | —    | 5.325           | 1.700                    |
| Dreverhoff, No. 331 ..      | 15        | —       | —    | 7.250           | 1.700                    |
| Laurent .. .. .             | 15        | —       | —    | 7.800           | 1.700                    |

#### Experiment III.

A Laurent filter of 15 c.m. diameter was filled with some of the same fresh water, containing 1.8 m.grms. of organic matter, ten separate times. 100 c.c. was put on one side after each filtration and estimated as before.

**Organic Matter obtained after the successive Filtrations.**

|              |       |               |       |
|--------------|-------|---------------|-------|
| First .. ..  | 5.350 | Sixth .. ..   | 2.225 |
| Second .. .. | 3.125 | Seventh .. .. | 2.050 |
| Third .. ..  | 2.975 | Eighth .. ..  | 1.875 |
| Fourth .. .. | 2.625 | Ninth .. ..   | 1.800 |
| Fifth .. ..  | 2.450 | Tenth .. ..   | 1.800 |

What can we conclude from these unexpected results? The following:—1. That organic matter is taken up by the passing of fresh or salt water through filters, whatever may be the kind of filter used. 2. That a filter-paper, after successive washings with the same water, loses less and less organic matter, but that it is necessary to wash it a large number of times to obtain constant and exact results.

We may draw the following practical conclusions from these results:—A fresh or salt water should never be filtered before analysis, but it must be left to settle completely (even for forty-eight hours), and a sufficient quantity to make the estimation removed with a pipette. Thus we shall not be liable to such errors as I have just pointed

out, and which might be of enormous importance in deciding on the potability or otherwise of a water, or on the possible contamination of sea-water at or near oyster-beds.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 3.

## TECHNICAL RESEARCH AND THE COLLEGE SYSTEM.

By W. P. DREAPER, F.I.C.

IN criticising the work of the technical colleges, many who are closely connected with their inner workings are, in spite of the familiarity which this involves, unable to answer the question so often propounded, How is it that they turn out so little original work? Assuming that these colleges are well organised, that their teaching staff is the best obtainable, that their fittings are perfect in their way, and that the stream of students through their lecture-halls is a continuous one, what is the result as measured by the original experimental work carried on in their laboratories? The professors in many of the colleges having a chemical department have answered this question recently ("Duty-free Alcohol," by T. Tyrer, *Journ. Soc. Chem. Ind.*, March, 1904). In answer to the query, "Would duty-free alcohol help research in your laboratory?" they have replied, "Yes, it would help research, but no industrial research is done." Probably realising the seriousness of this statement, one professor complained that the manufacturers did not bring up subject matter for such research, and another said that all research ultimately influenced industry in the long run.

The professors, if they knew more about the position of the manufacturers, would realise that they are leaning on a broken reed when they expect help from that quarter. The average manufacturer is, for the most part, incapable of indicating lines of research to the professor. His knowledge is of a business character, and it is not likely that he will be willing to see any experimental work connected with his factory routine more or less publicly carried on in the college laboratories, possibly under the eyes of the sons of his competitors. The alternative, viz., that the work shall be conducted in the professor's private laboratory does not meet the case. For here the professor is carrying on private work for his own convenience and profit. This, if carried out on any large scale, must seriously interfere with the success of the general college work.

The effect of this has been found so injurious that the authorities have even had to interfere on behalf of the students. The days when the professor was content to occupy a chair, give two lectures a week to the senior students, and spend the rest of his time in private research have happily gone, and can never return.

The only work the manufacturers could bring before the professors lies within the function of the works experimentalist, and is already part of the every-day routine.

The result of the present state of affairs is that the research conducted in the majority of technical laboratories runs on very narrow and circumscribed lines. The chemical department, for instance, will probably confine itself to so-called "organic" research, and this, now the famous diazo reaction has been worked to death, is apparently becoming increasingly difficult to initiate. With few exceptions this fairly represents the present-day state of affairs. This is generally acknowledged to be most unsatisfactory.

Many causes are put forward to explain this state of affairs; one is that the professors and demonstrators in the technical colleges are not men who have had the general and necessary technical experience to enable them to initiate such work. They lack the habit of picking up such lines of thought as comes from the constant and close contact with industrial operations on the large scale. Lacking this power of initiation they turn on the poor



manufacturer, who, trying his best to make 5 per cent on his capital, has little time to find answer to the question put to him, As you do not supply us with ideas for research what can you expect to find of advantage to the industrial world in our laboratories? Judging by the character and volume of the published research work carried on in these institutions, and from practical experience also, the position of the senior student is equally unsatisfactory. (This remark seems to apply still more to the United States). One or two of these may be engaged on research with the professor, but except in very rare cases the student himself will be unable to start any original work from want of experience. He will miss this important and necessary part of his training.

It is assumed that students in their last year of study cannot be better employed than in research connected more or less closely with their future career. In the case where students have not a specified line of future work cut out for them such research may give the necessary impetus in a specific direction.

The position of the technical man after leaving college and entering the industrial world is a definite one. He will experiment on a real and practical scale. He will find himself surrounded by a series of operations very varied in their way, and working on a large scale, and with practical objects seldom thought of in the college laboratory.

It is at this stage that the student's practical knowledge commences; although of course his college training is necessary ground-work for his subsequent operations. This I think is hardly realised by the professors on account of the inferior position which the technical branch is supposed to take when compared with the theoretical. In his daily round the technical man will see many operations working by rule-of-thumb. This means that the trained man has not yet stepped in to offer an explanation of their working. Many ideas will probably strike him, of a more or less theoretical nature, which will be quite outside his sphere of work as a practical man. It is taken for granted that he will be unable from force of circumstances to follow up these ideas connected with the theoretical side of his work. Beyond making a mental note of them, he must pass on to the pressing demands of the day. The opportunity is not his; the work must remain undone.

We have therefore, on the one hand, students starving for want of original work, and on the other, practical men unable to conduct such work rather from want of opportunity than ideas, and far above them all the professor sitting in his chair and railing at the manufacturer for not supplying him with ideas.

All this is the outcome of the position taken up by the professor. He has looked in the wrong direction for help.

The following scheme is put forward with the idea of overcoming this more or less serious state of affairs, and bringing the student more in touch with the problems he will have to face in his future life's work. These semi-theoretical, or even purely theoretical, problems arising out of the actual and practical experience of the experimentalist, are surely the very thing that "last year" students should be working on; under the present conditions this is impossible. It is not at all probable that these problems will be attacked by the technical man for want of opportunity. In this way many ideas lie dormant and unproductive.

The position of the teacher, student, and worker is therefore as follows:—The professor, from his want of practical knowledge, or want of an every-day experience in the practical matters of his profession, can only be expected to initiate research work of a purely theoretical nature, and this in doses as judged by the publication of such work. No man can, nor should be expected to, supply a department with ideas for research. A one source supply would also be one-sided in its nature. On the other hand, the professor from his position, past training, which we will assume to be of a high order, and from the time he is able to devote to general study, should be just the man to value in their early stages the ideas presented as subject-matter for re-

search. The professor is therefore made the corner stone of the scheme, which would work as follows:—

- (a). A research board would be founded in every technical or scientific college of repute, consisting of the professors and demonstrators.
- (b). Technical men who have passed through the college course (or others under certain conditions) would be invited by the Board to submit any ideas of a theoretical or semi theoretical nature which they think fit subject for research, suggested to them by their work, but owing to circumstances they are unable to carry out themselves.
- (c). The Board, after favourable consideration, would put a senior student in communication, through the Board, with the said technical man. The student, or even students, under such direction would commence a research on this special subject, carrying out the experimental part under the direction of the college staff under certain specified and general rules.
- (d). The results, if of a satisfactory nature, would be published under the name of the experimentalist. Special mention being made of the student and the college.
- (e). For the benefit of the general students research meetings would be held in class under the professor, where the aims and progress of the research would be indicated but not necessarily discussed.

In some such way as this the technical man would find a partner only too willing to engage in this kind of work. The staff control would guarantee the quality of the work done, and the Research Board would supply the connecting link between the student and the technical expert. The college should be in reality as in name a technical institution.

It might be urged that the relations of the manufacturer to the experimentalist would stand in the way of any such scheme. The experience I have collected goes to prove that this is not the case so long as the work is essentially theoretical in its nature or not too closely connected with the factory work.

For any such scheme to succeed it would be necessary that a general scheme for all colleges should be decided on, and that within certain limits outside experimentalists should be able to submit ideas to the college Board. This work, in addition to that already initiated by the staff, should afford in time sufficient material for an extended system of research, far-reaching in its scope, and also I think in its influence. It would tend to relieve the staff from the strain that it apparently is under in providing sufficient material for this purpose. It would bring the quantity of work done in this country into better comparison with that done in the European States without at the same time becoming trivial in its nature.

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Lecture Experiments for the Demonstration of Mass Action.—A. v. Dieterich and Lothar Wohler.—These experiments, which serve to illustrate mass action, are based upon the influence exercised by a solution of caustic potash on calomel:—1. With a solution of KOH at 1/100th the red colouration of phthalein disappears on agitation with calomel. 2. With a solution of KOH of a strength greater than 1/100th, we do not observe the decolouration of phthalein on agitation with Hg<sub>2</sub>Cl<sub>2</sub>. 3. With KOH at 1/100th, in the presence of chloride of potassium, we observe an analogous phenomenon. 4. If to a solution of potash, which has been decolourised by calomel, we add a few drops of a solution of KCl, the red colour reappears. 5. Equal parts of a solution of KOH at 1/100th and saturated KCl do not react on calomel. 6. If the red solutions obtained in experiments 2, 3, and 4 are heated, the colour disappears, and reappears on cooling.—*Zeit. Anorg. Chem.*, vol. xxxiv., p. 194.

THE DECOMPOSITION OF THE FINAL  
FRACTIONS OF MONAZITE INTO COMPONENTS,  
AND THE PREPARATION OF PURE  
GADOLINIUM OXIDE.\*

By R. MARC.

*Preparation of Gadolinium Oxide.*

GADOLINIUM was discovered in samarskite by Marignac in the year 1880, and was called  $Y_a$ . He separated it from the mixture of earths by purifying the nitrates, and separated it from the samarium which he called  $Y_\beta$  by fractionating with potassium sulphate. He characterised the oxide as having the highest atomic weight of those earths which resemble it in their behaviour towards potassium sulphate. Marignac's experiments were repeated by Lecoq de Boisbaudran, the existence of  $Y_a$  was established, and with Marignac's approval the name gadolinium was given to it. Crookes examined for cathode luminescence a gadolinium prepared by himself, and established a spectrum with three lines, one in the red and two in the orange. He concluded, from the spectroscopic behaviour, that gadolinium could be split up into three components. Bettendorf, by the use of the potassium sulphate method as modified by him, prepared pure gadolinium, and proved the elementary nature and purity by the constancy of the weights and the unaltered solubility of the separate fractions in saturated potassium sulphate solution. Benedicks recently obtained pure gadolinium oxide by crystallising the nitrates, and prepared a large number of its salts. He shares the opinion which Lecoq advocates, in opposition to Crookes, that the cathode luminescence of the gadolinium is caused solely by impurities.

For the preparation of the gadolinium oxide a material was placed at my disposal by Prof. W. Muthmann; it had been prepared by Herr L. Weiss from the final chromic acid fractionations of many kilogrammes of monazite. The last fractions were again treated with chromic acid, and then the final fractions again taken. This process was repeated some few times. The last fractions formed my material. It was of a decided yellow colour, and showed in the absorption spectrum the bands of the erbium components, samarium and neodymium, but no lines of praseodymium, even with the broadest layer. This material was treated as follows (*cf.* the tabular statement of the progress of the fractionation, Table I.).

After careful purification I possessed only 87 grms. altogether. It is clear that to separate the single components from such a small mass extraordinarily careful work was necessary. I was soon convinced that by the ordinary method of fractionation the material would in a short time be split into nothing but small portions before it was possible to bring about a definite separation.

Before, however, I pass to the details of the work, I will mention the methods of separation which I employed. These were the ammonia, the oxalic acid, and the potassium sulphate methods.

*The Ammonia Method.*

The fractionation with ammonia was performed as follows:—The whole of the material was divided into portions of 30 grms. Each such portion was dissolved in hydrochloric acid, diluted with two litres of water and accurately neutralised, and very dilute ammonia solution was dropped in from a separating funnel, while the liquid was kept in continual motion by means of a glass stirrer. The quantity of the ammonia was measured so that it corresponded to about half the quantity of oxide. The corresponding fractions of the separate portions of 30 grms. were again mixed and divided by means of ammonia into two parts, &c. By this method the erbium accumulated in the precipitates and the yttria in the liquids.

*The Oxalic Acid Method.*

Crookes employed oxalic acid for separating terbium (*Phil. Trans.*, clxxiv., 911). He dropped oxalic acid into the strongly acidulated boiling solution of the nitrates of the earths until a turbidity resulted, and then removed this turbidity by the addition of boiling nitric acid, and allowed to cool slowly. The oxalates then crystallise out in large crystals, which frequently enclose some mother liquor, and thus the separation is naturally delayed. To obviate this I proceeded as follows:—

The 10 per cent strongly acid solution of the nitrates was heated to boiling in a little flask, and rapidly boiling oxalic acid added until a permanent precipitate or turbidity began to form. Then the flask was cooled quickly, being vigorously agitated all the time, and the oxalates separated out as an exceedingly fine crystalline powder. The filtrate from the oxalates was concentrated to the original volume and again treated as described above.

The crystals which I obtained by this method were very small, and the results better than those obtained by Crookes's method, which I also employed for some experiments.

*The Potassium Sulphate Method.*

The potassium sulphate method served principally to separate didymium and samarium from the remaining earths. It was first used by Mosander. Afterwards Marignac (*Comptes Rendus*, xc., 899) investigated the solubility ratios of the double potassium sulphate salts in saturated solution of potassium sulphate.

Lecoq de Boisbaudran (*Comptes Rendus*, cii., 398) and Bettendorf (*Ann. d. Chem. u. Pharm.*, cclvi., 160) also used this method of separation, the latter in a somewhat modified form. I generally kept to Bettendorf's directions. But first of all I distinguished two methods, according to the aim I had in view:—

I. If I wanted to separate quickly a greater quantity of material into two parts, one of which would contain the greater part of the colourless earths and the other the greater part of the neodymium and samarium, then the following method was adopted:—

The neutral, or only very slightly acid, concentrated solution of the nitrates was introduced into a 4-litre flask, which was filled with a concentrated solution of potassium sulphate, the bottom being covered with crystals of this salt. The flask was filled up to the top with potassium sulphate solution, closed and sealed, and turned upside-down. This was done to prevent the settling of the precipitate formed on the bottom of the flask. The separation of the double salt begins at once. As soon as the liquid has cleared, which takes 1 to 2 hours, it is drawn off. With 4 litres of liquid about 10 grms. of the earths go into solution.

The precipitate was treated as is further described below, converted into nitrate, and then another 10 grms. brought into solution by the same method. This process was repeated as long as the oxide which went into solution showed no absorption streaks of neodymium and samarium, or only showed them very faintly. Thus the material had been divided into two parts, the first of which consisted of all the earths which had gone into solution, and the second of the part which had remained undissolved.

II. But when it was a question of obtaining from a material which contained neodymium and samarium the colourless earths, quite free from these two substances, disregarding the smallness of the results and the waste of time, the following method was used:—

According to the quantity of the material a 1 to 2 litre flask was only half filled with a concentrated solution of potassium sulphate. The bottom was covered with crystals, the concentrated neutral solution of the nitrates introduced into it, the flask closed and shaken on a shaking machine, with short interruptions, for two to three days. Almost the whole of the earth goes into the precipitate, and only 1 to  $\frac{1}{2}$  gm. remains in solution. The solution was, however, always quite free from neodymium and samarium,

\* From the *Zeitschrift für Anorg. Chem.*, xxviii., 121.

even if they were contained in the original material in considerable quantity. If the material contained no colourless earths, generally nothing, or only minute traces, went into solution. The treatment could be repeated until all the earths of the yttrium group were removed; but these operations, even with small quantities of material, would take months.

Concerning the treatment of the solutions and precipitates obtained, the following remarks must be made:—The solutions were simply treated with ammonia, the hydroxides precipitated were filtered off, dissolved, precipitated with oxalic acid, and the oxalates ignited. The precipitates of double sulphate could not be dissolved in water. Bettendorf (*loc. cit.*) treated them with hydrochloric acid, but it is necessary to use a very large quantity of acid and to heat for hours in order to bring them entirely into solution. For this reason I decomposed the double sulphates with sodium carbonate.

About an equal quantity of sodium carbonate and a little water were added to the double salts, and the mixture was heated to boiling for some time. By this means the double sulphate is completely converted into a basic carbonate, which again, for the most part, dissolves in the excess of sodium carbonate. Hydrochloric acid is then added until carbonic acid is no longer evolved; the earths are first precipitated as carbonates, and then dissolve in the excess of hydrochloric acid. If a residue remains still undissolved, this is again heated with soda, otherwise the earths are precipitated immediately from the hydrochloric acid solution by means of ammonia, and are filtered off, dissolved, precipitated with oxalic acid, and the oxalates ignited.

The whole quantity of the material at my disposal was treated with potassium sulphate by method I., in order to separate it into two groups, the one containing the yttria, erbium, terbium, and gadolinium, and the other the neodymium and samarium. Altogether four fractionations were necessary for this purpose. The four solutions together formed the first group and amounted to 45 grms.; the undissolved residue formed the second group and amounted to 42 grms.

The oxides of group I. showed only very faintly the bands of neodymium and samarium. They were decomposed into two parts by a series of ammonia fractionations, and of these two parts one did not show the slightest trace of the erbium spectrum, and the other no trace of neodymium or samarium. To avoid a loss of material I fractionated by the following method:—

The oxides were divided by means of ammonia into two equal parts, a precipitate and a filtrate, and precipitate and filtrate were each divided into two halves. In this way a series of four members was obtained. The two middle members were then further treated, and those parts which corresponded to the first or fourth members, as regards their spectra, were added to these members respectively. Thus the whole of the material was divided into two halves, separated by four fractionations. Each of these two halves was again divided. The extreme members were already separated by six fractionations, and the middle members were again treated till they were all added to the two extremes. Thus it was possible, by a series of 36 fractionations, to divide the material into two totally different parts without the least loss. The great number of fractionations necessary for such a separation is easily understood when we consider that the number of operations necessary to bring the inner members to the same state of purity as the outer is always greater the further the outer members are separated from one another.

The first thing to do was to find out in which part of the above mentioned separation the gadolinium would first be.

As the gadolinium is more strongly basic than erbium, it was to be assumed that it would be contained in the second part of the separation, which was shown by its spectrum to be free from erbium, and was far less yellow. This part was therefore again fractionated with ammonia.

according to the above scheme. Altogether about forty operations were performed, but in the last series of fractionations the separate members were not combined in two, but in three groups, according to the magnitude of their atomic weights. (In order to avoid converting from equivalent weights to atomic weights in dealing with mixtures, I have retained the expression atomic weight and calculated to the formula  $R_2O_3$ ).

The oxides with atomic weight 125—139 are in the first group. They were so largely adulterated with yttrium earths that it was worth while treating for gadolinium. There were altogether 10 grms. of yellow coloured oxides, which were almost free from absorption bands. The fractions 140—147 occurred in the middle group, amounting altogether to 10 grms. These oxides possessed a fainter yellow colour, and showed the samarium lines clearly and the neodymium lines faintly. The third portion showed the neodymium bands very strongly, and in addition, the samarium bands faintly. There were altogether 5 grms. of oxides of the greyish blue colour which is peculiar to neodymium oxide.

The high atomic weight of the second group leads us to infer the presence of a high percentage of gadolinium, as it could not, in any case, be caused by the comparatively small quantity of samarium. The gadolinium was thus adulterated in this group, on the one hand by yttria, and on the other by samarium and neodymium in small quantities. It was evident that it could not be easy to isolate in a pure condition one component from 10 grms. of such a mixture. Marignac's (*Comptes Rendus*, xc., 899) statements that yttria was soluble in less than 100 parts of potassium sulphate, gadolinium in about 200 parts, while samarium was only soluble in 400 parts, and neodymium was practically insoluble, led me to perform the separation with potassium sulphate, according to the method given under II.

The concentrated neutral solution of the nitrates was each time shaken vigorously for three days with 500 c.c. of concentrated solution of potassium sulphate, when the following amounts went into solution successively:—

| I. 0.46    | grm. oxide | Atomic weight, for $R_2O_3$ | 117.8  |
|------------|------------|-----------------------------|--------|
| II. 0.474  | "          | "                           | 121.15 |
| III. 0.624 | "          | "                           | 142.7  |
| IV. 0.472  | "          | "                           | 150.8  |
| V. 0.45    | "          | "                           | 153.7  |
| VI. 0.44   | "          | "                           | 154.78 |

(Free from neodymium and samarium).

The above numbers show that the solubility of the gadolinium in a saturated solution of potassium sulphate is considerably less (about one-tenth) than that stated by Marignac (*loc. cit.*). The reason for this is that on standing at rest strongly supersaturated solutions are formed. I hope to be able to communicate more accurate investigations of this point soon.

All the oxides of this series were quite free from absorption spectra. The first members were decidedly yellow, the last only faintly. The last member with atomic weight 154.78, resembled gadolinium most closely, the following figures being given by the various authors as its atomic weight:—Marignac, 156.75; Lecoq de Boisbaudran, 155.9; Cleve, 155; Bettendorf, 156.33; Benedicks, 156.38. My preparation was doubtless still adulterated with small quantities of yttria.

The residue (7 grms.), which did not go into solution with potassium sulphate solution, was again subjected to treatment with the same solvent. But it was not possible to bring more substance free from samarium into solution. However, I succeeded in completely removing the small quantities of neodymium by once applying potassium sulphate method I., and was also successful in removing the greater part of the samarium. From the material (3 grms.) thus obtained, which contained a small amount of samarium, I was able, by a long series of exceedingly carefully performed ammonia separations, to obtain  $\frac{1}{2}$  gm. of

oxide free from samarium, and determine its atomic weight. The determinations gave:—

- I. 0.2201 grm. of oxide gave  
0.3666 grm. of sulphate.

Difference 0.1465 grm.; hence atomic weight = 156.3.

- II. 0.2444 grm. of oxide gave  
0.4070 grm. of sulphate.

Difference 0.1626 grm.; hence atomic weight = 156.35.

These two numbers agree satisfactorily with the values 156.33 and 156.38, given by Bettendorf and Benedicks.

After strong ignition gadolinium oxide is only faintly yellow. It is difficult to say definitely whether this suggestion of a yellow colour is characteristic of gadolinium oxide or is due to the admixture of traces of a coloured earth. But it is a fact that a specimen of gadolinium which does not show this colouration has not yet been obtained. The colour disappears in a current of hydrogen.

The salts, and the solutions of the salts of gadolinium, are colourless. The solutions show no absorption spectrum. Under the influence of the cathode rays neither pure gadolinium sulphate nor oxide becomes luminescent, and no cathode luminescence spectrum is shown. (Cf. Baur and Marc, *Ber. Deutsch. Chem. Ges.*, xxxiv, 2460-66).

There can hardly be any further doubt concerning the elementary nature of gadolinium. If gadolinium were a mixture, neighbouring earths with atomic weight higher than 156 would be known to us. However, this is not the case, the atomic weight 156 of gadolinium being, as Marignac has already noticed, the highest of the earths standing near it.

The atomic weight determinations were performed by Krüss's method (*Zeit. Anorg. Chem.*, iii., 44-59), by the conversion of the oxides into sulphate. Previous to the determination the oxides were carefully purified.

The chief impurities were platinum, iron, calcium, aluminium. The oxides were therefore dissolved in acid. The excess of the latter was neutralised with ammonia, and sulphuretted hydrogen was passed in for 6 hours. After filtering and concentrating the solution, it was precipitated with ammonia in presence of ammonium chloride, filtered, washed, dissolved, again precipitated with ammonia, thoroughly washed, again dissolved and finally precipitated from a slightly acid solution with the purest oxalic acid, the oxalate being thoroughly washed and ignited. These operations were not always sufficient to remove the last traces of iron. Hence, if the solution of the oxides still gave iron reactions, the hydrochloric acid solution was concentrated until it began to crystallise. The iron remained in solution when all the earths had crystallised out.

0.2 to 0.4 grm. of the purified oxides were then heated in a porcelain crucible until the weight became constant, in order to ensure the absence of carbonic acid and moisture. These oxides were then very carefully moistened with two or three drops of water, and enough dilute hydrochloric acid was added to dissolve them. Then they were heated over a small flame on an iron plate about 5 m.m. thick until they were completely dissolved. 1 to 2 c.c. of dilute sulphuric acid were then added carefully by means of a small pipette, and the liquor evaporated on the iron plate over a very small flame till the sulphate began to crystallise. When crystallisation began the flame was raised. When no more acid fumes came off the flame was brought close up under the iron plate. After some time the crucible was placed on a pipeclay triangle and heated over an open flame, which was at first small, but was afterwards gradually raised, without, however, being allowed to approach within 2 or 3 c.m. of the bottom of the crucible. Heating for 4 to 6 hours was generally sufficient, and a second weighing was almost always constant.

#### Samarium.

For the preparation of samarium I had the choice of the neodymium preparations, rich in samarium, or the middle

preparations, less rich in samarium but quite free from neodymium. I decided to use the latter, as it is far more difficult to separate samarium from neodymium than from erbium, and it was important for me to get a preparation quite free from neodymium.

The material for the preparation of the samarium consisted of only 10 grms. of atomic weight 125 to 139. It possessed a well-marked yellow colour; its spectrum showed, besides decided quantities of samarium, small quantities of erbium, but no neodymium. The separation of the samarium from the erbium is performed comparatively easily.

Ammonia was used for the separation, and I arranged so that only as much ammonia was added as corresponded to 1 grm. of the oxide. Even after the first three fractionations the remaining liquid appeared quite free from erbium. To make quite sure two more fractionations were performed, and the remaining 5 grms., which were quite free from erbium, were treated in such a manner that those oxides which showed the samarium spectrum most strongly were united, purified, and further fractionated. After rather a long series of fractionations I finally obtained, as the richest in samarium, 0.2 grm. of a yellow preparation, which was quite free from absorption bands of foreign earths.

It was interesting to observe whether this samarium oxide, when mixed with lime or yttria like the three other earths with absorption bands, would give a cathode luminescence spectrum. This was, however, not the case.

The cathode spectrum characteristic of neodymium and erbium was indeed faintly visible, and, as in the case of yttria, it may safely be stated that a complete elimination of this spectrum is hardly possible, owing to the sensitiveness of the method.

The absorption spectrum of my samarium preparation showed the two following bands:—

$\lambda$  487-475 and  $\lambda$  467-462,

while Lecoq de Boisbaudran gives as the middle of the samarium bands (*Berichte Deutsch. Chem. Ges.*, xxxv., 2382):—

$\lambda$  480     $\lambda$  463     $\lambda$  417     $\lambda$  400.75.

I could not see either of the two last.

From the material on the right-hand side by repeated fractionation with ammonia I could also get preparations very rich in samarium, but these retained the last traces of neodymium with extraordinary energy.

I also succeeded in preparing from the extreme fractions 9 grms. of almost perfectly pure neodymium, which were adulterated with only very small quantities of samarium.

The left-hand side is very interesting. By alternate fractionations with ammonia and oxalic acid, first erbium and then holmium ( $\lambda$  454-449) could be separated, and finally the brown ochre terbium preparations were obtained with the bands  $\lambda$  464-461, which are probably characteristic of terbium, and of which I gave an account some time ago (*Ber. Deutsch. Chem. Ges.*, xxxv., 2382).

This work is an unpublished part of a more extensive piece of work which I have been doing for two years in Prof. W. Muthmann's laboratory in the Technical High School at Munich. Its chief aim is to examine the usefulness and application of methods of separation of the rare earths, methods which made it possible to separate from a relatively very small quantity of material single components in a pure state (neodymium, samarium, gadolinium), and to increase the proportion of others so that it was possible to make observations of their properties (terbium).

**Application of Blondlot Rays to Chemistry.**—Albert Colson.—The author's experiments show that the Blondlot rays are probably due to the formation of basic salts. This kind of molecular condensation produces analogous effects to those which are obtained by mechanical compression.—*Comptes Rendus*, cxxxviii., No. 15.

# PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, April 20th, 1904.

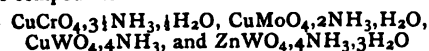
Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

(Concluded from p 224).

### 65. "Ammoniacal Double Chromates and Molybdates." By SAMUEL HENRY CLIFFORD BRIGGS.

A number of double salts of ammonium chromate belonging to a series of compounds having the general formula  $M_2^{II}M^{II}(RO_3)_2 \cdot 2NH_3$  have already been described (*Trans.*, 1903, lxxxiii., 391). Other members of the same series have now been prepared,  $M^{II}$  being either  $NH_4$  or  $K$ ;  $M^{II}$  being  $Cu$ ,  $Zn$ ,  $Cd$ ,  $Co$ , or  $Ni$ , and  $R$  indicating either  $Cr$  or  $Mo$ . The corresponding tungstates could not be obtained.

The compounds—



have been incidentally prepared during the investigation.

### 66. "The Hexahydrated Double Chromates. Magnesium and Nickel Compounds." By SAMUEL HENRY CLIFFORD BRIGGS.

The double chromates  $M_2^{II}M^{II}(CrO_4)_2 \cdot 6H_2O$ , in which  $M^{II}$  is magnesium or nickel and  $M^{II}$  is  $K$ ,  $Rb$ , or  $Cs$ , have been prepared.

The nickel compounds were obtained by adding a solution of the alkali chromate to a solution of nickel acetate, at the ordinary temperature for the rubidium and caesium salts, and at  $6^\circ$  for the potassium compound.

The magnesium compounds crystallised out when cold concentrated solutions of their components were mixed, the preparation of the potassium salt being carried out at  $-10^\circ$ .

Both the potassium compounds are very efflorescent at the ordinary temperature, whilst in the case of the series of salts in which  $M^{II}$  is the same, the stability increases with the atomic weight of the alkali metal. This variation is in the same sense as that found by Tutton (*Trans.*, 1896, lxi., 521) for the relative stabilities of the corresponding double sulphates.

### 67. "Hydrocellulose." By CHARLES FREDERICK CROSS and EDWARD JOHN BEVAN.

The residues described by Stern (*Trans.*, 1904, lxxxv., 336) having the empirical composition of cellulose are no doubt products of hydrolysis and reversion, and are constitutionally different from the original cellulose; but they are in no case identical with those described by Girard, and therefore this investigator's exhaustive account of the actions of acids on cellulose remains unaffected by the criticisms contained in the above-mentioned paper. The statements (*loc. cit.*) as to the causes of the attendant structural changes are, moreover, improbable.

Cellulose is a chemically labile and structurally plastic aggregate, occupying an intermediate position between the two extremes, determined by the action of (a) alkali hydroxides, (b) the halogen hydracids, both in presence of water.

It is suggested that the terms hydracellulose and hydro-cellulose respectively may for the present be retained to designate the two groups of derivatives obtained by the processes (a) and (b).

### 68. "Bornylcarbimide." By MARTIN ONSLOW FORSTER and HERBERT MOORE ATTWELL.

The appearance of a paper by Doht and Haager (*Monatsh.*, 1903, xxiv., 844), dealing with the production of phenylcarbimide by the action of nitrous acid on phenylcarbamide, leads us to record a similar observation which we have made in connection with bornylcarbamide.

Bornylcarbimide,  $C_{10}H_{17}N:C:O$ , prepared by adding solid sodium nitrite to bornylcarbamide nitrate suspended

in water at  $0^\circ$ , is a colourless, crystalline substance which melts at  $72^\circ$ ; it is very volatile, and the vapour is intensely pungent. A 2 per cent solution in benzene has  $[a]_D +46.5^\circ$ . It is hydrolysed by acids and alkalis, yielding bornylamine, whilst aniline converts it into bornylphenylcarbamide, previously obtained from bornylamine and phenylcarbimide (*Trans.*, 1898, lxxiii., 393).

Dibornylthiocarbamide,  $S:C(NH \cdot C_{10}H_{17})_2$ , obtained on heating bornylamine with carbon disulphide and alcohol until hydrogen sulphide is no longer liberated, melts at  $227^\circ$ .

Bornylamine bornyldithiocarbamate,—



is produced at the same time, and melts at  $78^\circ$ .

Bornylamine thiocyanate crystallises from hot water in long, lustrous needles melting at  $178^\circ$ ; above this temperature, it changes into dibornylthiocarbamide.

### 69. "Reduced Silicates." By CHARLES SIMMONDS.

The substance left when lead silicates are reduced by heating in hydrogen (*Trans.*, 1903, lxxxiii., 1449) is in general shown to be a compound which can be regarded as a combination of the metal and silica, in the same sense as the original silicate is a combination of the metallic oxide and silica. In some cases, however, a certain proportion of metallic lead is mixed with the substance; this occurs when the original silicates (for example, orthosilicates and basic silicates) contain a greater number of basic than of acidic oxides in the silicate molecule.

The reduced residues are generally more refractory than the original silicates. Treatment with the commoner acids and oxidising agents has little effect on them, but they are decomposed by hydrofluoric acid and by fusion with alkali carbonates. The term "silicites" is suggested for these reduced silicates.

Similar results were obtained with the silicates of copper, iron, nickel, and cobalt.

### 70. "Picryl Derivatives of Urethane and Thiourethane." By JAMES CODRINGTON CROCKER and FRANK HAROLD LOWE.

The authors show that the reaction between picryl chloride, thiocyanates, and alcohols is due to the formation of the  $\psi$ -thiourethanes,  $Pin:C(SH) \cdot OX$ , as intermediate products, which subsequently react with picryl chloride, and pass into the picriminothiocarbonates,  $Pin:C(SPi) \cdot OX$ . The results obtained with the cyanates, picryl chloride, and alcohol confirm this view. Under these conditions, the corresponding urethanes,  $PinH \cdot CO \cdot OX$ , are formed, and in the case of ethyl alcohol, the picriminocarbonate,  $Pin:(OEt) \cdot OPi$ , is also produced. The following compounds are described:—

Picryl isopropyl picriminothiocarbonate, melting at  $147^\circ$ , and the corresponding amyl ester (m. p.  $138.5^\circ$ ), picrylmethylurethane ( $192^\circ$ ), picryl-ethylurethane ( $147^\circ$ ), picryl-n-propylurethane ( $139^\circ$ ), picryl-isopropylurethane ( $177.5^\circ$ ), picryl-isobutylurethane ( $134^\circ$ ), picrylamylurethane ( $131^\circ$ ), picryl ethyl picriminocarbonate ( $222^\circ$ ). Picryl thiocyanate is a crystalline powder which does not melt, but decomposes on heating.

The urethanes are tautomeric, the solutions in associated solvents being coloured, whilst those in non-associated solvents are colourless.

### 71. "The Oxime of Mesoxamide (isoNitrosomalonomide) and some Allied Compounds. Part III. Tetra-substituted Derivatives." By MARTHA ANNIE WHITELEY.

In a previous communication (*Trans.*, 1903, lxxxiii., 43) reference was made to isonitrosomalondimethylanilide,  $C_{17}H_{17}O_3N_3$  (m. p.  $109^\circ$ )—a tetra-substituted derivative of isonitrosomalonomide—and to a compound (m. p.  $192^\circ$ ), described as isomeric with it, which was obtained together with the corresponding ketone (m. p.  $172^\circ$ ) by the action of nitrosyl chloride on malondimethylanilide. Although the examination of this abnormal reaction is still incomplete, the present note is put forward in consequence of the

appearance of a paper on isonitrosomalondimethylamide by Ratz (*Monatsh.*, 1904, xxv., 55).

The compound melting at  $192^\circ$  (mol. wt. in naphthalene = 311) has the formula  $C_{17}H_{15}O_3N_3$  or  $C_{17}H_{17}O_3N_3$ , and on reduction with zinc dust and acetic acid or with aluminium amalgam, yields a compound,  $C_{17}H_{17}O_2N_3$  or  $C_{17}H_{19}O_2N_3$ , which crystallises in colourless, prismatic needles, and melts at  $185$ – $186^\circ$ . When treated with alcoholic potassium or sodium hydroxide, it forms crystalline alkali salts, and from their solutions mineral acids precipitate a crystalline acid,  $C_9H_8O_3N_2$ , melting with decomposition at  $256^\circ$ , which yields an *ethyl ether*,  $C_9H_7O_3N_2Et$  (mol. wt. in naphthalene = 222), crystallising in octahedral prisms, and melting at  $168^\circ$ .

*Aminomalondimethylanilide*,  $(CO \cdot NPhMe)_2CH \cdot NH_2$ , prepared by reducing isonitrosomalondimethylanilide with zinc dust and acetic acid, forms highly refractive prisms melting at  $108^\circ$ .

*Nitromalondimethylanilide*,  $(CO \cdot NPhMe)_2CH \cdot NO_2$ , obtained together with the compound melting at  $192^\circ$  by the action of fuming nitric acid on isonitrosomalondimethylanilide dissolved in chloroform saturated with nitrosyl chloride, forms colourless prisms melting with decomposition at  $156^\circ$ , yields crystalline, yellow alkali salts, and is readily reduced by zinc dust and acetic acid to mesoxdimethylanilide. When boiled with piperidine in alcoholic solution, nitromalondimethylanilide yields a white crystalline compound melting at  $184^\circ$ , probably *dihydroxymalondimethylanilide*,  $(CO \cdot NPhMe)_2C(OH)_2$ , which has also been prepared from isonitrosomalondimethylanilide by a similar method or by the action of potassium hypobromite.

*Malontetraphenylamide*,  $(CO \cdot NPh)_2CH_2$ , prepared by the action of diphenylamine on malonyl chloride, crystallises in colourless prisms melting with decomposition at  $219$ – $220^\circ$ . The isonitroso-derivative,  $(CO \cdot NPh)_2C \cdot N \cdot OH$ , forms pale yellow prisms melting with decomposition at  $237$ – $238^\circ$ . The potassium salt,  $(CO \cdot NPh)_2C \cdot N \cdot OK$ , forming slender, yellow needles, gives with ferrous sulphate the rich bluish-purple precipitate characteristic of this group of oximes, and regenerates the isonitroso-compound on treatment with mineral acids or carbon dioxide. The acetyl derivative (pale yellow prisms, m. p.  $190^\circ$ ), the benzoyl derivative (colourless prisms, m. p.  $175^\circ$ ), and the ethyl ether (m. p.  $164$ – $165^\circ$ ) have been prepared. The amino-derivative,  $(CO \cdot NPh)_2CH \cdot NH_2$ , crystallises in colourless prisms, and melts at  $200$ – $201^\circ$ .

*Nitromalonanilide*,  $(CO \cdot NHPh)_2CH \cdot NO_2$  (m. p.  $141^\circ$ ), is obtained together with a compound melting at  $128^\circ$  by the action of fuming nitric acid on isonitrosomalonanilide dissolved in chloroform saturated with nitrosyl chloride. The amino-derivative,  $(CO \cdot NHPh)_2CH \cdot NH_2$ , forms white scales melting at  $141$ – $142^\circ$ .

Malonmonophenylamide,  $NHPh \cdot CO \cdot CH_2 \cdot CO \cdot NH_2$ , crystallises with  $\frac{1}{2}H_2O$ , and, when anhydrous, melts at  $153$ – $154^\circ$  (Freund gives  $163^\circ$ ); the isonitroso-derivative,  $NHPh \cdot CO \cdot C(CO \cdot NH_2) \cdot NOH$ , forms needles melting at  $180$ – $181^\circ$  with decomposition.

The investigation of the derivatives of isonitrosomalondimethylamide is being continued.

#### THE FARADAY SOCIETY.

An Ordinary Meeting was held on May 9th, 1904, at the Institution of Electrical Engineers, Mr. BERTRAM BLOUNT in the Chair.

Dr. CHARLES E. FAWSITT read a Paper entitled "*Studies in Viscosity.*"

*Some Relations of Viscosity to Salt Formation.*—The viscosity of a mixture of two or more substances in aqueous solution is equal to the product of the viscosities which the solutions of the substances separately have. If, however, the substances act chemically on one another then the viscosity of the mixture is different from the calculated value. By numerical illustrations from cases where a strong acid is neutralised by a strong base, this

difference is shown to have a constant value for equimolecular quantities. This value corresponds to the formation of undissociated water from the hydrogen and hydroxyl ions. The difference between the calculated and observed values, in the case where a weak base is mixed with a strong acid, has a value which varies with the base in the case of urea, and its derivative appears to vanish.

*Viscosity as an Additive Property.*—A comparison of the viscosity constants of some urea derivatives and aurides of the fatty series shows that, for a difference in the composition of  $CH_2$ , there is a corresponding difference in the viscosity constant. There is, however, a pronounced "constitutional" effect, as shown by the different values obtained for isomeric substances.

Mr. B. BLOUNT referred to the difficulties in making accurate and reliable viscosity measurements on which important deductions are based.

Dr. T. M. LOWRY discussed the relationship between viscosity and conductivity, and conductivity and temperature; and he showed how, in the case of very dilute solutions, the conductivity-temperature curves converge to zero conductivity at  $-39^\circ C.$ , just as the viscosity curve for pure water does.

Dr. RUDOLF observed that with different forms of apparatus different results are obtained.

Dr. FAWSITT, in reply, said that his results were all obtained with one apparatus, and that they were strictly comparable. Referring to Dr. Lowry's remarks, he added that viscosity is inversely proportional to migration velocity.

Dr. F. M. PERKIN then read a Paper by A. FONTANA and himself on "*The Electrolytic Oxidation of Anthracine.*"

The authors have taken up the study of the oxidation of anthracine, primarily to ascertain whether it was possible to obtain a good laboratory method for the preparation of anthraquinone. The first attempts were made with solutions in acetone, platinum electrodes being employed. Although oxidation took place in solutions of anthracine in acetone, it was not found possible to oxidise more than about 55 per cent of the anthracine. Attempts were then made to electrolyse anthracine suspended in 20 per cent sulphuric acid, or in caustic alkali, to which an oxygen carrier had been added. Various carriers were employed, the most satisfactory being chromium, cerium, or manganese salts. The anode consisted of a lead vessel; the cathodes, four in number, were also of lead and were placed in four small porous cells. There was also a lead paddle, which was connected with the positive pole. The total anode surface was about 6 square decimetres; current density, 1 ampère per square decimetre; temperature,  $70$ – $80^\circ C.$ ; and E.M.F. from 2.5 to 3.5 volts. The yield of anthraquinone was about 10 per cent, and it was not found possible to increase the yield more than 1 or 2 per cent above this. It was afterwards found that almost equally good results were obtained without a diaphragm. In this case the stirrer,  $\frac{1}{4}$  square decimetre in area, was made the cathode. In discussing the results of their experiments, the authors consider that, owing to its volatility and the comparatively poor yield, a solution in acetone cannot be recommended. On a large scale no method in which a diaphragm is employed would be satisfactory, owing to the migration of the  $SO_4^{--}$  ions from the cathode to the anode side. They then briefly review the patents of the Höchst Farben Fabrik, Darmstädter, and Martin Moest. Le Blanc criticised Darmstädter's patent and doubted whether it would be possible to obtain any appreciable quantity of anthraquinone owing to the reduction of the chromate by the cathodic hydrogen. The authors are of the opinion that to a certain extent the chromium, manganese, and cerium salts act as chromium does when employed in the electrolytic bleach industry, viz., by prevention of reduction. Moest claims, in his patent, to obtain 100 per cent yields of anthraquinone. The authors have been unable to obtain much more than 80 per cent; they are, however, of the opinion that very likely better results would be obtained on a manufacturing scale.

Mr. BLOUNT suggested the use of thallium salts. He thought the energy bill in this instance might not necessarily be an all important item.

Dr. STEINHART thought better results would be obtained if the anode solution were removed from the cathode.

Mr. INGLIS discussed the effect of the potential difference between the anode and solution.

Mr. GASTER thought the cost of energy was of prime importance. He suggested the use of petroleum as a base for colours instead of tar.

## OBITUARY.

### PROF. A. W. WILLIAMSON, F.R.S.

It is with great regret we record the death of Prof. A. W. Williamson, which occurred on Friday last, May 6, at his residence at Hindhead.

Prof. Williamson had only just completed his eightieth year; his health had been gradually failing for the past two or three years, but it is only about a month ago that his condition began to cause any anxiety.

With his death another of the most prominent early workers in modern chemistry is lost to us; in fact, of the six Past Presidents of the Chemical Society who had been Fellows for half a century, to whom a banquet was given on November 11, 1898, one only, Dr. William Odling, is left. The other five, Sir Joseph Gilbert, Sir Edward Frankland, Sir Frederick Abel, Dr. J. H. Gladstone, and now Prof. A. W. Williamson, have joined the great majority.

Dr. Williamson was born on May 1, 1824, and after some years of chiefly private education, he studied at the Universities of Heidelberg and Giessen, under Gmelin and Liebig. At Giessen he published his first chemical researches. He afterwards spent three years in Paris, studying the higher mathematics. In 1849, he was appointed Professor of Practical Chemistry in University College, London, and in 1855 Professor of Chemistry in the same College, still retaining the chair of Practical Chemistry. Soon after his first appointment at University College, Prof. Williamson published his researches on "Etherification and the Constitution of Salts," for which important and successful work he was awarded the Royal Medal of the Royal Society. In this research he struck at the very root of the chemical problems connected with atomic and molecular weights, and realised and cleared up those mysterious modes of explanation, such as contact or catalytic action, which before his time were always introduced when no other explanation was forthcoming; in fact, the chemistry of our time would not be the science that it is but for the work of Williamson. He was also one of the first to introduce the idea of dynamics into chemical science, and his suggestion of the dynamical theory of the voltaic battery, and of dynamic mobility in apparent stability, has been extremely fruitful in more recent years.

Among the more important papers written by Prof. Williamson we may call attention to the "Dynamics of the Galvanic Battery," in 1864; one on "The Classification of the Elements in relation to their Atomicities," and one on "Chemical Nomenclature and Notation"; all three in the same year.

In the year 1865, Prof. Williamson made an investigation which is of peculiar interest at the present time; this was on the "Composition of the Gases Evolved by the Bath Spring called the King's Bath," and he read a paper bearing this title at the Birmingham meeting of the British Association in that year. The composition of these gases was found to be over 96 per cent of nitrogen, about 3 per cent of carbonic acid, 0.5 to 0.6 per cent of oxygen, and about 0.2 per cent of marsh-gas. In conjunction with Dr. J. W. Russell, he wrote a paper on "A New Method of Gas Analysis," and in the year 1881, as President of the

Chemical Section at the York Meeting of the British Association, he delivered an Address on "The Growth of the Atomic Theory."

Prof. Williamson was elected a Fellow of the Chemical Society in 1848, and was twice President, in 1863 and 1869. He was elected Fellow of the Royal Society in 1855. In 1873 he was President of the British Association at Bradford, and was elected Treasurer in the following year. In 1875 the Royal Academy of Science at Berlin elected him a corresponding member of the Section of Physics and Mathematics, and in the same year he was appointed a member of the Senate of the University of London, in which institution he took an active part in promoting the establishment of degrees of Science. In 1887 he resigned his Professorship at University College, and was elected Emeritus Professor. He was a member of many learned societies both at home and abroad, and held the Honorary Degree of LL.D. of the Universities of Dublin and Edinburgh. In 1855 he married the third daughter of the late Prof. T. Hewitt Key, who survives him; he also leaves a son and a daughter.

## NOTICES OF BOOKS.

*Descriptive Chemistry.* By LYMAN C. NEWELL, Ph.D. In Two Parts. London: D. C. Heath and Company. 1904. Pp. 590.

This book is conveniently divided into two parts, each part forming a separate volume. Part I. contains the text of the facts, laws, and theories of chemistry, and Part II. contains the experiments. The text has been selected and arranged with special reference to the requirements of teachers as well as of students, and the experiments have been prepared to meet the needs of schools in which the time available for laboratory work is short.

The book is well got up and printed, but though published in England its origin is undoubtedly American, as is shown by the quaint spelling. An excellent feature is the good index; this covers no less than 70 pages of two columns each, and is of the most comprehensive character.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 15, April 11, 1904.

**Electric Osmosis in Methyl Alcohol.**—A. Baudouin. —Electric osmosis is quite perceptible in methyl alcohol, but considerably less than in water under the same conditions. A difference of potential of 250 to 300 volts is necessary, as against 60 to 100 for water. The first experiments were made with absolute alcohol, but the author afterwards found that the presence of a small quantity of water did not materially affect the results.

**Calculation of the Heat of Combustion of Nitrated Organic Compounds.**—P. Lemoult.—In various former publications the author shows that the heat of combustion of organic compounds,  $C_xH_yOp$ , can be calculated by utilising the formula—

$$Z = 102x + \frac{55}{2}y - \Sigma p\rho + A.$$

The same method may be adopted for compounds which contain nitrogen, by using the new conventions,—

$$f(c - az) = \frac{1}{2}f(c^* - az^*) = \frac{1}{2}f(c^* \equiv N) = 31 \text{ cals.},$$

and—

$$f(az - H) = 23 \text{ cals.}$$



These formulæ give satisfactory results, as can be shown by the 140 cases examined.

**A New Method of obtaining Calcium Carbide.**—L. M. Bullier.—The author draws attention to the fact that in 1895 he described a method for preparation of calcium carbide founded on the same principles as that described by M. Moissan in a recent paper.

**Estimation of Nitrogen.**—Léon Débourdeaux.—The author describes a new method of estimating nitrogen, founded on the action of the alkaline monosulphides on nitrogen compounds in presence of various salts, chiefly the alkaline hyposulphites. The reaction takes place in a bulb communicating with a modified Schloësing's apparatus. The ammonia produced is received in excess of pure hydrochloric acid, and can be estimated accurately by weighing. This method, although limited directly, can be applied to a general estimation of nitrogen after having modified the radicle containing the nitrogen by adding phenol or acid functions, or by attaching the nitrogen group to a radicle containing a phenol or acid function.

**Influence of Hydriodic Acid on the Oxidation of Sulphurous Acid.**—A. Berg.—Hydriodic acid, according to the quantity present, retards or accelerates the oxidation of sulphurous acid. For each solution of this latter there seems to exist a proportion of hydriodic acid which has no influence on the oxidation. For 4 per cent solutions the proportion is 3 per cent—corresponding to rather less than 1 molecule of hydriodic acid to 3 of sulphuric acid. Hydriodic acid is not the only body which accelerates the oxidation of sulphurous acid. The same is true of manganese chloride and ferrous chloride. This is a similar effect to their oxidising influence on organic substances. Soluble metallic iodides act in the same way. On the contrary, chloride and bromide of potassium have no action. Finally, hydrochloric acid retards the oxidation, and when strong may even annul it altogether.

**Chloruration of Phenyl Carbonate in presence of Iodine.**—Et. Barral.—The author has previously described the preparation of a neutral bichlorated phenyl carbonate,  $\text{CO}(\text{OC}_6\text{H}_4\text{Cl})_2$ , by chloruration of phenyl carbonate in presence of iodine, and also the formation of neutral pentachlorophenyl carbonate,  $\text{CO}(\text{OC}_6\text{Cl}_5)_2$ , by the reaction of carbon oxychloride on pentachlorophenol. He further investigated the possibility of obtaining all the chlorine derivatives of phenyl carbonate, either by direct chloruration in presence of energetic chlorinating agents, or by synthesis by means of carbon oxychloride and the chlorophenols. By chloruration in presence of iodine, of aluminium chloride, or antimony pentachloride, and by varying the method of operation, the author now succeeds in obtaining all the degrees of chlorination of phenyl carbonate. He also establishes the fact that the chloruration of phenyl carbonate can be effected in presence of anhydrous ferric chloride.

*Bulletin de la Société Chimique de Paris.*  
Series 3, Vol. xxix., No. 14.

**Production of Crystallised Salts of Bismuth.**—A. de Schulten.—Already inserted in full.

**A Particular Property of some Hydrated Salts.**—A. de Schulten.—Already inserted in full.

**A Method for the Crystallisation of slightly Soluble Bodies.**—A. de Schulten.—Already noticed.

**The Arsenides of Copper.**—Albert Granger.—Already inserted in full.

**The Estimation of Vanadium in its Alloys.**—Paul Nicolardot.—Already noticed.

**History of the Acetals of the Polyatomic Alcohols of the Sugar Series.** New Researches on the conditions of Combination of Mannite and of Paraldehyde. —J. Meunier.—Early in his research the author found that ethylic aldehyde and its product of condensation, par-

aldehyde, gave a compound with mannite as easily as benzoic aldehyde; an analogous compound was also furnished by valerianic aldehyde, but much more slowly. One of the characteristic properties of these new bodies is that of decomposing on boiling with acidulated water, regenerating the constituent aldehyde and polyatomic alcohol. The aldehyde being volatile is carried off by the steam, and thus separated from the alcohol which remains in solution. As, on the other hand, these acetals are insoluble in the medium in which they are formed, we have here the principle of a method of extraction for the alcohols derived from the sugars, which has long been wanted. The author then goes on to describe a number of experiments with natural mannite, showing the influence on the results of the duration of the reaction, and varying the proportions of mannite, paraldehyde, and hydrochloric acid.

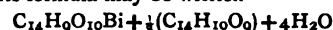
**Volumetric Estimation of the Lime and Magnesia simultaneously present in Solutions of Chloride of Sodium.** (Waters from Salt Marshes). —Alex. d'Anselme.—Already inserted in full.

**On Benzene-ortho-nitrobenzylic Alcohol and its Transformation into Phenylindazol.**—P. Freundler.—Already noticed.

**The Compounds of Bismuth and Tannin.**—Paul Thibault.—During the research the author has been carrying on for some time past, on the compounds of bismuth with the organic acids having phenolic functions, he found it impossible to eliminate, by washing, the mineral acids retained by the compounds of bismuth prepared by double decomposition, or by the precipitation of a salt of the metal by a concentrated solution of the body just used, so he tried another method. Hydrated oxide of bismuth was placed in contact in the cold with a slight excess of tannin; after three days the supernatant liquor no longer contained any tannin: a fresh quantity was added, and after a very prolonged contact (three months), after having made sure that an excess of tannin was present, the material was washed and dried at a low temperature. The product obtained is a yellow amorphous powder similar to bismuthogallic acid, soluble in the mineral acids, soda, and potash, insoluble in the alkaline carbonates and bicarbonates, as well as in neutral solvents. Analysis gives the figures:—

|              | Calculated for $(\text{C}_{14}\text{H}_9\text{O}_9)_3\text{Bi}_2$ . |     |       |       |
|--------------|---------------------------------------------------------------------|-----|-------|-------|
| C .. ..      | 33.64                                                               | and | 33.54 | 33.82 |
| H .. ..      | 2.79                                                                | "   | 2.83  | 2.81  |
| Bi .. ..     | 27.35                                                               | "   | 27.90 | 27.91 |
| O (by diff.) | 36.22                                                               | "   | 35.73 | 35.46 |

Therefore its formula may be written—



without defining either the number or the place of the substituted oxyhydriles. To sum up, tannin only acts on the hydrated oxide of bismuth and not on the anhydrous oxide; the bismuth is substituted for the phenolic hydrogen, leaving the acid function free; tannin or digallic acid in excess, when acting on oxide of bismuth, becomes fixed to the bismuthotannin formed, in a proportion varying from  $\frac{1}{2}(\text{C}_{14}\text{H}_{10}\text{O}_9)$  to  $\frac{1}{4}(\text{C}_{14}\text{H}_{10}\text{O}_9)$ , according to the duration of the time of contact, and to obtain the product  $\text{C}_{14}\text{H}_9\text{O}_{10}\text{Bi}$  only the theoretical amounts of the component parts should be placed in contact.

**The Phenolic Urethanes of Piperidine.**—M. Bouchetal de la Roche.—The author gives the properties of some new chlorised, bromised, and nitro-urethanes. These bodies were prepared with the object of identifying them with the bodies he expected to obtain directly by reacting with chlorine, bromine, and nitric acid on phenylic urethane. He gives the results of the action of these agents on different urethanes.

**The Constitution of the Colouring-matter of Indigo.**—L. Maillard.—The hypothesis of two substances,  $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_2$ , makes possible the existence of at least nine bodies of the formula  $\text{C}_{32}\text{H}_{20}\text{N}_4\text{O}_4$ , while only two are known. The theory of the two indogenides of isatin

requires further the previous superoxidation of half the indoxyl, while the other half remains unchanged, and, finally, the dislocation of the pyrrolic nucleus to transform the blue indigo into the red. On the other hand, the author's theory, admitting only a single body  $C_{16}$ , and two  $C_{32}$ —that is to say, just the number of bodies known—accounts for the facts in a much more simple manner.

**Action of Sulphur and Selenium on the Bromide of Phenylmagnesium and the Bromide of  $\alpha$ -Naphthylmagnesium.**—M. Taboury.—The action of oxygen on these organo-magnesium compounds, having been found by M. Bodroux to give a certain number of phenols, the author wished to see whether reactions of a similar character could be obtained with the other metalloids of the same family, leading to the formation of thio-, seleno-, and telluro-phenols. The results of his experiments, which are described at length, show that the expected results were obtained. Following this up, he found that by acting directly on the compound  $Mg\langle\begin{smallmatrix} Br \\ X \end{smallmatrix}\rangle R$  with an acid chloride, it was possible to obtain the ethers of these thio-phenols and seleno-phenols.

**Note on the Use of a New Denaturising Agent for Commercial Alcohols.**—M. Cari-Mantrand.—Already inserted in full.

**The Detection of the Products of the Contamination of Water by Methyl Sulphurous Violet and Sulphonised Paradiabenzene.**—H. Causse.—Already noticed.

**Pressure Regulator for Fractional Distillations under Reduced Pressure.**—M. Gab. Bertrand.—This paper requires the accompanying diagram to make its meaning clear.

**Separator for Fractional Distillation under Reduced Pressure.**—M. Gab. Bertrand.—This paper also requires its accompanying diagram.

**A New Refrigerating Agent.**—MM. Braconnier and C. Chatelain.—Already noticed.

**Apparatus for the Estimation of Nitrogen.**—R. Marquis.—Requires the accompanying diagram.

## MISCELLANEOUS.

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 9th inst., The Duke of Northumberland, K.G., President, in the Chair. The Chairman announced that he had nominated the following Vice-Presidents for the ensuing year:—Sir William Abney, K.C.B., Mr. Shelford Bidwell, Right Hon. Lord Kelvin, Dr. Ludwig Mond, Sir Thomas H. Sanderson, G.C.B., Sir Felix Semon, Sir James Crichton-Browne (Treasurer), and Sir William Crookes (Honorary Secretary). The following were elected Honorary Members:—Prof. E. H. Amagat, Prof. L. P. Cailletet, Prof. J. M. Crafts, Prof. E. W. Morley, Prof. E. C. Pickering, Prof. and Madame Curie, Prof. H. L. Le Chatelier, Prof. G. Lippmann, Prof. J. W. Bruhl, Prof. G. H. Quincke, Prof. E. Fischer, Prof. F. W. G. Kohlrausch, Prof. H. Landolt, Prof. L. Boltzmann, Dr. H. Kammerlingh Onnes, Prof. H. A. Lorentz, Dr. G. Lunge, Prof. P. T. Cleve, and Prof. P. Zeeman. The special thanks of the Members were returned to Mrs. Frank Lawson and Sir Thomas Sanderson, G.C.B., for their donations to the Fund for the Promotion of Experimental Research at Low Temperatures. The following were elected Members:—Mr. P. M. Brophy, Mr. W. J. Canter, Mr. Eyre Crowe, Mr. H. T. Ellis, Miss E. Morgan, Mr. Berkeley Portman, and Mr. Burnett Tabrum, J.P.

**Institute of Chemistry Examinations.**—Pass Lists (April, 1904).—*Final A.I.C. Examination—Branch "A" (Mineral Chemistry)*: James John Ellis, B.Sc. (Vict.),

Yorkshire College, Leeds; Arthur Gordon Francis, B.Sc. (Lond.), Royal Coll. of Science, London, and under S. R. Trotman, F.I.C., and in the Government Laboratory; James Henry Pizey, Assoc. R.C.Sc. (Lond.), Royal Coll. of Science, London, and under Bertram Blount, F.I.C.; Louis John Ezekiel Riley, Univ. of Edinburgh, Glasgow and W. of Scot. Tech. Coll., and King's Coll., London. *Branch "B" (Metallurgical Chemistry)*: Reginald Tyler, Univ. Coll., Nottingham, and under A. H. Allen, F.I.C. *Branch "D" (Organic Chemistry)*: Reginald William Lane Clarke, Finsbury Tech. Coll., London; Oliver Charles Minty Davis, B.Sc. (Lond.), Univ. Coll., Bristol; Bernard Farmborough Howard, Finsbury Tech. Coll., London. *Branch "E" (Analysis of Food and Drugs, and of Water, including Examination in Therapeutics, Pharmacology, and Microscopy)*: Harold Frederick Barke, The Owens Coll., Manchester, and under A. E. Parkes, F.I.C.; Cyril Dickinson, B.Sc. (Vict.), Yorkshire Coll., Leeds, under J. Kear Colwell, F.I.C., and W. Scott Tebb, M.A., M.D., A.I.C.; Ernest Garratt, B.Sc. (Vict.), Univ. Coll., Liverpool, and under J. Campbell Brown, D.Sc., F.I.C., and W. Collingwood Williams, B.Sc., F.I.C.; Charles Edgar Male, School of the Pharmaceutical Society, King's Coll., London, and under Thomas Tickle, F.I.C.; John Robert Stubbs, M.Sc. (Vict.), Univ. Coll., Liverpool, and under J. Campbell Brown, D.Sc., F.I.C., and W. Collingwood Williams, B.Sc., F.I.C.

**The Formation of Alloys of Sodium and their Importance in the Phenomena of Cathodic Polarisation.**—M. Sack.—Cathodes of lead and of tin are polarised in soda solutions, and the rapid disengagement of hydrogen corresponds with their swelling up, a result caused by the intermediate formation of alloys of sodium on these cathodes. The alloys of sodium with lead and tin, obtained by the fusion of the metals in a porcelain or iron crucible in the presence of hydrogen, have properties varying according to their proportion of the alkaline metal. Alloys of lead with 40 or more atoms per cent of sodium, as well as those of tin with more than 25 per cent, are reduced to powder on contact with water, at the same time giving off hydrogen. It is to the formation of such alloys that we must attribute the attack of the electrodes and their polarisation. The disengagement of hydrogen takes place on lead with an electromotive force of 0.7 volt, and on tin with 0.4 volt. The polarisation of the cathodes take place at potentials of 1.5 and 1.4 volts respectively. Alloys of lead and tin, in alcoholic solution of chloride of lithium cooled down to  $-80^{\circ}$ , have potentials very close to those observed with lead and tin alone. The alloys  $Pb_2Na + Pb_x$  and  $Sn_3Na + Sn_x$  in the cold give the lowest potentials, very close to those of lead and tin, while the alloys  $Pb_2Na + Na_x$  and  $Sn_3Na + Na_x$  give higher potentials. A protective layer is formed on the cathodes which does not occur at the ordinary temperature. When a protective layer is not formed on the alloys poor in the alkaline metal, at the ordinary temperature, we observe a remarkably constant potential, although their composition varies by reason of the disengagement of hydrogen (diminution of the amount of sodium). Measurements made with these electrodes give the same results as those obtained with electrodes of lead or zinc when polarised. Experiments with mercury led to analogous conclusions; with amalgams poor in sodium we obtain a potential identical with that furnished by mercury alone, while amalgams richer in sodium than  $Hg_3Na$  diverge in a most distinct manner. At a low temperature, we still observe the formation of a protective layer on the cathode. With platinum and zinc we still observe the swelling of the electrodes, but they do not become reduced to powder. The swelling of the platinum, like that of lead, is much more marked in acid than in alkaline solutions. These facts explain easily why the precipitated sodium soaks into the metallic cathode less easily than the hydrogen, and shows directly that the disengagement of hydrogen at these electrodes is a secondary phenomenon.—*Zeit. Anorg. Chem.*, vol. xxxiv., p. 286.

**Accurate Volumetric Apparatus.**—Messrs. John J. Griffin and Sons, Ltd., inform us that they are now able to supply volumetric apparatus tested at the National Physical Laboratory. For many purposes for which these vessels are used a certificate of the exact value is not needed; it is sufficient to know that the graduations are correct within some specified limit of error. This knowledge is secured by placing the Laboratory Mark on vessels which come up to the specification. The plan used for limits of error is the one adopted by the "Reichsanstalt," and it is proposed for the future to adopt it at the National Physical Laboratory.

**Technics.**—This magazine, which is intended as an aid to Technical Progress, is maintaining the promise of the earlier numbers. No. 5, which is the current number, contains articles on Paper Making (Esparto), Boot and Shoe Manufacture, Structural Design, Technical Education in America, the Formation of Loops and the Construction of Looped Fabrics, and many others of technical and general interest. Practically all the articles are illustrated, and of these this number contains nineteen, besides reviews, personal items, *Technics* competitions, &c. *Technics* is published by Messrs. George Newnes, Ltd., and is well worth reading, both by manufacturers and others engaged in the various technical industries.

## MEETINGS FOR THE WEEK.

**TUESDAY, 17th.**—Royal Institution, 5. "Meteorites," by L. Fletcher, F.R.S., &c.  
Society of Arts, 8. "Pewter and the Revival of its Use," by Lasenby Liberty.

**WEDNESDAY, 18th.**—Chemical, 5.30. "Action of Nitrosyl Chloride on Pinene," by W. A. Tilden. "The Electrolytic Estimation of Minute Quantities of Arsenic," by H. J. S. Sand and J. E. Hackford. "The Decomposition of the Alkyl Ureas," by C. E. Fawcett. "The Action of Sodium Methoxide and its homologues on Benzophenone Chloride and Benzal Chloride" (Part II.), by J. E. Mackenzie and A. F. Joseph. "The Formation of Periodides in Nitrobenzene Solution—II., Periodides of the Alkali and Alkaline Earth Metals," by H. M. Dawson and Miss E. E. Goodson. Microscopical, 8. "Note on Grayson's Rulings," by E. M. Nelson. Exhibition of Flower Seeds under Microscopes, by C. Beck.

**THURSDAY, 19th.**—Royal Institution, 5. "Great Britain and Europe (1763–1793)," by Arthur Hassall, M.A.

**FRIDAY, 20th.**—Royal Institution, 9. "The Radiation and Emanation of Radium," by Prof. E. Rutherford, F.R.S.

**SATURDAY, 21st.**—Royal Institution, 3. "Sonata Style and the Sonata Forms" (with Musical Illustrations), by Donald Francis Tovey, B.A.

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### CONTENTS.

| ARTICLES:—                                                                                                                                | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------|------|
| The Connection between the Volatility of Compounds and the Chemical Forces at Play within the Molecule, by Geoffrey Martin, B.Sc. (Sond.) | 241  |
| London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.                                                              | 242  |
| A Practical Method for the Estimation of Ozone, by A. Brunck                                                                              | 243  |
| PROCEEDINGS OF SOCIETIES—                                                                                                                 |      |
| ROYAL SOCIETY.—Conversations at Burlington House                                                                                          | 245  |
| CHEMICAL SOCIETY                                                                                                                          | 246  |
| PHYSICAL SOCIETY                                                                                                                          | 248  |
| NOTICES OF BOOKS                                                                                                                          | 249  |
| CORRESPONDENCE                                                                                                                            | 250  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                                                     | 251  |
| MISCELLANEOUS                                                                                                                             | 251  |
| MEETINGS FOR THE WEEK                                                                                                                     | 252  |

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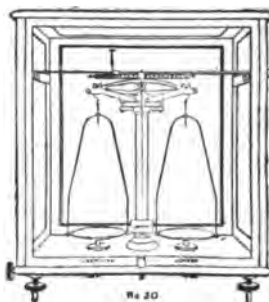
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2321.

## THE CONNECTION BETWEEN THE VOLATILITY OF COMPOUNDS AND THE CHEMICAL FORCES AT PLAY WITHIN THE MOLECULE.

By GEOFFREY MARTIN, B.Sc. (Lond.).

In the CHEMICAL NEWS for January 29, 1904 (vol. lxxxix., p. 41), I showed that there exists an intimate connection between the volatility of the fluorides, chlorides, and oxides, and the intensity of the chemical forces which the fluorine, chlorine, and oxygen atoms are capable of exerting.

I propose to give here a brief abstract of a somewhat laborious investigation on this point which I have been engaged in for some time past.

1. Chemically unstable compounds are, as a class, characterised by their volatility and fusibility; chemically stable compounds by their involatility and infusibility.

For example,  $\text{SiCl}_4$ ,  $\text{BCl}_3$ ,  $\text{AlCl}_3$ ,  $\text{SnCl}_4$ , &c., are all easy to volatilise and all easy to decompose. The corresponding oxides,  $\text{SiO}_2$ ,  $\text{B}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SnO}_2$ , are hard to volatilise and hard to decompose. Indeed, viewed as a class, the metallic chlorides generally are easier to volatilise and easier to decompose than the oxides and fluorides.  $\text{AgI}$  is less stable than either  $\text{AgCl}$  or  $\text{AgBr}$ . It is also more volatile.  $\text{Ni}(\text{CO})_4$ ,  $\text{Fe}(\text{CO})_5$ , are characterised by their volatility and by the ease with which they can be decomposed.  $\text{UO}_3$ ,  $\text{MoO}_3$ ,  $\text{WO}_3$ ,  $\text{CrO}_3$ ,  $\text{Mn}_2\text{O}_7$ ,  $\text{OsO}_4$ ,  $\text{RuO}_4$  are the most volatile and fusible, and at the same time the most unstable oxides of U, Mo, W, Cr, Mn, Os, and Ru.  $\text{UCl}_5$  is easier to volatilise and easier to decompose than  $\text{UCl}_4$ .  $\text{WCl}_6$  is more volatile and less stable than  $\text{WCl}_4$ ;  $\text{MoCl}_5$ , than  $\text{MoCl}_4$ ;  $\text{MoBr}_4$ , than  $\text{MoBr}_3$ ; and  $\text{MoBr}_3$ , than  $\text{MoBr}_2$ ;  $\text{FeCl}_3$ , than  $\text{FeCl}_2$ ;  $\text{CrCl}_3$ , than  $\text{CrCl}_2$ ;  $\text{InCl}_3$ , than  $\text{InCl}_2$ ;  $\text{InCl}_2$ , than  $\text{InCl}$ ;  $\text{GaCl}_3$ , than  $\text{GaCl}_2$ ;  $\text{SbCl}_5$ , than  $\text{SbCl}_3$ , &c.

The compounds of mercury can be all easily volatilised, and at the same time can be all easily decomposed by heat. The same applies to the ammonium compounds. On the other hand, the compounds of Na and K can neither be easily volatilised nor decomposed by heat in this way.

$\text{MgO}$ ,  $\text{CrO}$ ,  $\text{SrO}$ , and  $\text{BaO}$  are characterised by their great stability and their great volatility;  $\text{BN}$  is involatile and stable;  $\text{C}_2\text{N}_2$  is volatile and unstable; the oxides of chlorine, iodine, nitrogen, are all easy to volatilise and easy to decompose;  $\text{ICl}$ ,  $\text{IBr}$ ,  $\text{ICl}_3$ ,  $\text{IBr}_3$ ,  $\text{Se}_2\text{Cl}_2$ ,  $\text{Se}_2\text{Br}_2$ ,  $\text{SeCl}_4$ ,  $\text{SeBr}_4$ ,  $\text{S}_2\text{Cl}_2$ ,  $\text{SCl}_4$ , &c., are all volatile bodies, and at the same time easy to decompose.  $\text{HF}$  is much more stable than  $\text{HCl}$  or  $\text{HBr}$ ; it is also much less volatile.  $\text{HF}$  is a liquid at ordinary temperature, whereas the others are gases.

The vast majority of the carbon compounds are either fluids at ordinary temperatures or become so within a range of  $150^\circ$ . It is the fusibility and volatility of the carbon compounds that is their most striking characteristic; but they are also extremely unstable under the action of heat. Indeed, the whole domain of organic chemistry is destroyed at a red heat.

Similarly the nitrogen compounds are characterised by their volatility and the ease with which they decompose under the action of heat. On the other hand, the silicates, borates, phosphates, metallic carbides, &c., are characterised by their great involatility and their great stability under the action of heat.

*Inference.*—From these facts we conclude that, statistically considered, there is a connection between the intensity of the

force with which atoms are bound together in the molecule and the volatility of the compounds—the connection being of such a nature that in general the more strongly attracted together are the atoms in the molecule (and therefore the more stable the molecule), the more strongly attracted together are the molecules themselves, and consequently the more involatile the compound.

2. High-grade valency compounds are usually volatile and fusible, low grade compounds involatile and infusible.

There exists a remarkable connection between the valency of an element and the volatility of the compounds to which it gives rise.

Compounds of a high valency grade are usually characterised by their volatility and fusibility, characteristics which become the more conspicuous the higher the type of combination we deal with.

In this connection we may recall to mind the conspicuous volatility of such compounds as  $\text{Ni}(\text{CO})_4$ ,  $\text{OsO}_4$ ,  $\text{RuO}_4$ ,  $\text{Mn}_2\text{O}_7$ , which are all remarkable by reason of their great volatility and fusibility, and because they are the highest types of combination known.

We will briefly recapitulate the chief facts bearing on the matter:— $\text{SbCl}_5$  is more volatile than  $\text{SbCl}_3$ ;  $\text{UCl}_5$  is more volatile than  $\text{UCl}_4$ ; and  $\text{UCl}_4$  more volatile than the lower chlorides;  $\text{WCl}_6$  and  $\text{WCl}_5$  are more volatile than  $\text{WCl}_4$ , and this more volatile than the still lower chlorides.  $\text{FeCl}_3$  is more volatile than  $\text{FeCl}_2$ ;  $\text{InCl}_3$ , than  $\text{InCl}_2$ ;  $\text{InCl}_2$ , than  $\text{InCl}$ ;  $\text{GaCl}_3$ , than  $\text{GaCl}_2$ ; and  $\text{GaCl}_2$ , than  $\text{GaCl}$ ;  $\text{CrCl}_3$  is more volatile than  $\text{CrCl}_2$ .

|                |                                                     |
|----------------|-----------------------------------------------------|
| $\text{CrO}_3$ | is more volatile than any of the lower oxides of Cr |
| $\text{MoO}_3$ | " " " " " " " $\text{Mo}$                           |
| $\text{WO}_3$  | " " " " " " " $\text{W}$                            |
| $\text{UO}_3$  | " " " " " " " $\text{U}$                            |
| $\text{OsO}_4$ | " " " " " " " $\text{Os}$                           |
| $\text{RuO}_4$ | " " " " " " " $\text{Ru}$                           |

$\text{Mn}_2\text{O}_7$  is a liquid, whereas all the lower oxides are involatile solids (cf.  $\text{MnO}_2$ ,  $\text{Mn}_2\text{O}_3$ ,  $\text{Mn}_3\text{O}_4$ ,  $\text{MnO}$ , &c.);  $\text{NO}_3$  is a liquid which does not freeze in a mixture of ice and salt (Berthelot);  $\text{N}_2\text{O}_5$  is a solid.

All the high-grade compounds of Cr, W, Mo, U, Va, &c., are characterised by their volatility. This tendency is particularly manifest in the multitude of high-grade oxyhalides they produce. The lower grade compounds of the same type are all much less volatile.

High-grade compounds are usually unstable, and it might be thought that this is the reason of their volatility and fusibility (see Part I.).

It certainly does not arise invariably from the greater molecular weight of the lower compounds.

For example,  $\text{SCl}_4$  has (as shown by vapour density) a molecular weight of 174, and  $\text{S}_2\text{Cl}_2$  a molecular weight of 135, yet  $\text{SCl}_4$  is much more unstable than  $\text{S}_2\text{Cl}_2$ .  $\text{SF}_6$  boils at  $-55^\circ$ ,  $\text{SO}_2\text{F}_2$  at  $-52^\circ$ ,  $\text{SOF}_2$  at  $-32^\circ$ , though their molecular weight, as shown by their vapour densities, stand in the ratio 146 : 102 : 86.  $\text{UCl}_5$  is more volatile than  $\text{UCl}_4$ , though, as shown by their vapour densities, the molecular weights stand in the ratio 417 : 382. The same applies to  $\text{InCl}_3$  and  $\text{InCl}_2$ ,  $\text{FeCl}_3$  and  $\text{FeCl}_2$ ,  $\text{GaCl}_3$  and  $\text{GaCl}_2$ ,  $\text{CrCl}_3$  and  $\text{CrCl}_2$ , &c., the vapour densities of all of which have been studied.

The reason of the difference in volatility seems to be that the molecules of the lower grade compounds attract each more strongly than the molecules of the higher grade bodies, and so it comes about that the lighter molecules are less volatile than the heavier molecules. It will be remembered in organic chemistry, unsaturated compounds are less volatile than saturated compounds of the same molecular weight.

3. Compounds which react in a very similar way with different reagents usually approach each other closely as regards volatility.

Chemically similar compounds usually approach each other closely as regards volatility. For example,  $\text{HCl}$ ,

HBr, HI are all chemically similar and all volatile enough to be gaseous at ordinary temperatures, though the molecule of HBr is more than twice as heavy as the molecule of HCl, and that of HI nearly four times as heavy.

Similarly,  $\text{BCl}_3$ ,  $\text{SiCl}_4$  are both volatile liquids;  $\text{SiO}_2$ ,  $\text{B}_2\text{O}_3$ , both fixed solids, &c. The general truth of this observation must be apparent to everyone who has even a superficial knowledge of chemistry.

Now the chemical similarity of two compounds arises from the identity of the intensity of the chemical forces at play within the molecule. For example,  $\text{SiCl}_4$  and  $\text{BCl}_3$  are chemically similar because:—

- The B and the Si attract Cl with nearly the same intensity of force.
- The B and the Si attract other radicles also with nearly the same intensity of force.

For when the first condition is fulfilled, any influence which is capable of removing the Cl from the one molecule will be capable of removing it from the other; and when the second condition is fulfilled, the B and Si will enter into the same reactions. So that the two molecules  $\text{BCl}_3$  and  $\text{SiCl}_4$  will, when subjected to the same influences, react in the same way. That, as a matter of fact, in  $\text{BCl}_3$  and  $\text{SiCl}_4$  the Cl is attached to the B and the Si atoms with nearly the same intensity of force appears from the thermal data:—

$$\frac{1}{2}[\text{B}, \text{Cl}^*] = 34.6 \quad \frac{1}{2}[\text{Si}, \text{Cl}^*] = 39.4,$$

and that B and Si attract not only chlorine but other radicles, such as oxygen, with nearly the same force appears from the data:—

$$\frac{1}{2}[\text{B}, \text{O}^*] = 52.8 \quad \frac{1}{2}[\text{Si}, \text{O}^*] = 52.7,$$

and that chemically similar atomic species in general owe their chemical similarity to the fact that they, in general, attract the same radicles with nearly the same intensity of force likewise appears from a study of thermo-chemical data (I hope shortly to publish a paper on this subject). For example, cobalt and nickel attract the same radicles with nearly the same intensity of force:—

| Reaction.                                                               | Co. | Ni  |
|-------------------------------------------------------------------------|-----|-----|
| $\text{R} + \text{Cl}_2 + \text{Aq} \dots \dots \dots$                  | 95  | 94  |
| $\text{R} + \text{Br}_2 + \text{Aq} \dots \dots \dots$                  | 73  | 72  |
| $\text{R} + \text{O} + \text{Aq} \dots \dots \dots$                     | 63  | 61  |
| $\text{R} + \text{O}_2 + \text{SO}_2 + n\text{H}_2\text{O} \dots \dots$ | 163 | 163 |
| $\text{RCl}_2 + \text{Aq} \dots \dots \dots$                            | 18  | 19  |

R stands for either nickel or cobalt; the numbers are the heat evolved by the corresponding reactions (Thomsen).

The fact that chemically similar compounds possess nearly the same degree of volatility and fusibility is almost certainly connected with the fact that the chemical forces at play within their molecules are of nearly the same intensity.

It seems therefore that it is the internal chemical forces which the atoms exert on each other in the molecule which decides the external attractive force with which the molecules themselves are attracted together, and therefore the volatility of the compound.

The mere magnitude of the molecular weight has very little influence as compared with the influence of the chemical forces in deciding the degree of volatility. And this is the reason why compounds composed out of very light molecules are often far less volatile than compounds composed of enormously heavier molecules, because the light molecules attract each other with a greater intensity of force than the heavy molecules do.

For example, the molecules of  $\text{OsO}_4$  are eleven times heavier than the Na molecules, and yet are enormously more volatile. Compare also H and He, O and F, Hg and Mg,  $\text{SiF}_6$  and  $\text{SOF}_2$ , &c.; in all these cases the heavier molecules are the most volatile. In fact, the influence of the molecular weight on the volatility only becomes apparent when we eliminate the effect of the chemical forces by considering chemically similar compounds (*i.e.*, compounds in which the chemical forces are of nearly the same intensity).

When we compare chemically dissimilar compounds (*i.e.*, compounds in which the chemical forces at play are of very different intensities), the influence of the molecular weight entirely disappears, and the chemical forces alone decide the volatility.

For example, the replacing of hydrogen in a molecule by Cl or F (atoms 35.5 and nineteen times heavier than H) sometimes leads to an increase of volatility, although the molecular weight is thereby greatly increased (Henri, *Rec. Trav. Chim.*, 1897, xvi., 218, 225), *e.g.*:—

|                                                                    |                                                       |
|--------------------------------------------------------------------|-------------------------------------------------------|
| $\{\text{CHCl}_3 \text{ boils at } 61^\circ$                       | $\{\text{CH}_2\text{Cl}_2 \text{ boils at } 41^\circ$ |
| $\{\text{CFC}_3 \text{ " " } 24^\circ$                             | $\{\text{CHFCl}_2 \text{ " " } 14.5^\circ$            |
| $\text{NC} \cdot \text{CH}_2\text{Cl} \text{ boils at } 123^\circ$ |                                                       |
| $\text{NC} \cdot \text{CHCl}_2 \text{ " " } 112^\circ$             |                                                       |
| $\text{NC} \cdot \text{CCl}_3 \text{ " " } 83^\circ$               |                                                       |

**Conclusion.**—We therefore conclude that the volatility of any given compound depends almost entirely upon the intensity of the chemical forces with which the atoms which compose it attract other atomic species, and that did we only know the exact intensity of these forces, and the law governing the relation between them and the intensity of the resultant molecular attraction, it would be possible to calculate mathematically the volatility of the compounds, a slight correction being necessary for the gravitational influence of the magnitude of the molecular weight.

Considerations similar to those which lead us to regard the volatility as depending upon the intensity of the chemical forces, lead us to believe that the chemical forces determine also the solubility, fusibility, hardness, &c., of compounds, so that these forces being known the chief chemical and physical properties of the compounds would also be (theoretically) deducible.

Elements are only compounds in which the atoms of the molecule are of the same kind, *e.g.*,  $\text{O}_2$  and  $\text{O}_3$  are two different compounds composed of atoms of the same kind. Consequently, the same laws which apply to compounds in general apply to elements in particular. Therefore the volatility, fusibility, solubility, &c., of elements themselves in general depend mainly upon the intensity of the forces with which they attract other atomic species, and to a very much smaller extent, upon the magnitude of the molecular weight. A knowledge of these forces would, in fact, give us a very complete picture of all the chemical and physical properties of the elements.

University of Kiel.

## LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING APRIL 30TH, 1904.

By SIR WILLIAM CROOKES, F.R.S.,  
and  
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, ESQ., M.Inst.C.E.,  
Water Examiner, Metropolis Water Act, 1871.

London, May 9th, 1904.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 195 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from April 1st to April 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 195 samples examined by us during the month, all were clear, bright, and well filtered.

The deficit in rainfall observed during March has been continued during April. The actual amount of rain registered at Oxford was 0.81 inch; the average rainfall for the month is 1.61 inches; thus we have a deficit of 0.80 inch. This, subtracted from the previous excess of 1.78 inches, leaves an excess for the year of 0.98 inch, or 14.0 per cent above the thirty-five years' average.

Our bacteriological examinations of 354 samples taken during the month have given the results recorded in the following table. Besides these samples we have examined 352 others from special wells, standpipes, &c., making 706 samples in all:—

|                                                                                                                 | Microbes<br>per c.c. |
|-----------------------------------------------------------------------------------------------------------------|----------------------|
| New River, unfiltered (mean of 24 samples) ..                                                                   | 109                  |
| New River, filtered (mean of 69 samples) ..                                                                     | 15                   |
| Thames, unfiltered (mean of 23 samples) ..                                                                      | 4798                 |
| Thames-derived water from the clear-water<br>wells of eight Thames-derived supplies (mean<br>of 190 samples) .. | 19                   |
| Ditto ditto .. .. . highest                                                                                     | 136                  |
| Ditto ditto .. .. . lowest                                                                                      | 0                    |
| River Lea, unfiltered (mean of 24 samples) ..                                                                   | 150                  |
| River Lea, from the East London Water Com-<br>pany's clear-water wells (mean of 24 samples)                     | 7                    |

Of the 283 daily samples taken from the general filter wells of the Metropolitan Water Companies, seventeen samples, or 6.0 per cent, were sterile. Only five samples, or 1.7 per cent, contained more than 100 microbes, and of these, none contained as many as 150 microbes per c.c. The five excess samples contained an average of 123 microbes per c.c. In March ten excess samples contained an average of 148 microbes per c.c. Thus it will be seen that, chemically and bacteriologically, the general supply is excellent.

We are, Sir,

Your obedient Servants,  
WILLIAM CROOKES.  
JAMES DEWAR.

## A PRACTICAL METHOD FOR THE ESTIMATION OF OZONE.

By A. BRUNCK.

Some years ago I remarked (*Berichte*, 1900, vol. xxxiii., p. 183a) that the practical methods of estimating ozone could only be based on titration with hyposulphite of iodine set free in a solution of iodide of potassium. Quite recently, Ladenburg (*Berichte*, 1903, p. 115) has examined several other methods for its estimation, based on the use of sulphite and arsenite of sodium, and has found that they give results much less accurate than those obtained by the iodide of potassium method.

It is well understood that ozone behaves very differently with iodide of potassium, according to whether the latter is in acid or neutral solution; that is to say, according as it is in the presence of iodine or of hydriodic acid. For equal quantities of ozone, and with  $N_2$  solutions, the amount of iodine set at liberty is 50 per cent higher in the former than in the latter case.

Chemical literature gives no information on this point; some writers state definitely that the ozone must be made to act on a neutral solution of iodide; others, on the contrary, recommend its previous acidulation to prevent the formation of iodate.

In most cases no importance seems to be attached to this question.

However, in a recent paper, R. Luther and H. Ingliis

(*Zeit. Phys. Chem.*, 1903, vol. xliii., p. 203) state that Brodie (*Phil. Trans.*, 1872, vol. clxii., p. 455) observed long ago that ozone reacts very differently with iodide of potassium, according to the conditions of the experiment. Brodie's work has been evidently passed over and forgotten, as it is never mentioned in any of the ordinary text-books, and Englers himself does not mention it in the monograph he has written on ozone.

It is very difficult to determine under which circumstances (in neutral or acid solution) the reaction of ozone on iodide takes place normally. As a matter of fact, on the one hand there was no method of estimation, free from all doubt, until quite lately; and on the other hand, it is not possible to prepare a gaseous mixture containing a known quantity of ozone. In face of this difficulty I consider that hydriodic acid gives the most exact results; it appears, in fact, that the reaction is extremely simple, and that the only cause of error is the direct oxidation of the hydriodic acid by the atmospheric oxygen. Now, direct experiment has shown me that this oxidation was absolutely negligible at the strengths at which my experiments were carried out. On the contrary, the action of the ozone on neutral solutions of iodide, seems as if it ought to be much more complex on account of the formation of free alkali, which might bring about secondary reactions and losses of ozone.

Later on, Ladenburg, and R. Quasig (*Berichte*, 1901, vol. xxxiv., p. 1184) described a method for estimating ozone by direct weighing. A glass bulb, closed with a tap, is weighed first full of pure oxygen, then full of ozonised oxygen; the difference of these two weights, under the same conditions of temperature and pressure, enable us to calculate the weight of the ozone contained in a known volume of the gas under consideration. Though this method appears to be so simple, yet it is difficult to apply in practice, as many minute precautions must be observed in carrying it out. In spite of this drawback, pointed out by the authors themselves, the method is valuable, as by its means we can check all the others; and, more particularly, we can solve the question of the action of ozone on iodide of potassium.

The numerous experiments made by Ladenburg and Quasig in this direction, have led to an altogether unexpected result. To obtain results agreeing with those furnished by the ponderable method, it is necessary to react with the ozone on a neutral solution of iodide, and only to acidulate the latter at the moment of titration.

I have confirmed my previous experiments by means of the ponderable method, and I am convinced that the conclusions arrived at by Ladenburg are perfectly correct, although they are contrary to all expectations.

The principle of the iodometric method of estimating ozone being known, I directed my attention to its practical application. With this object I constructed a special apparatus, similar to that proposed by W. Hesse (*Lehrbuch der tech. Gasanalyse*, C. Winkler, Third Edition, p. 119), for the estimation of carbonic acid in gases containing only a small proportion. It is constructed entirely of glass, in such a manner as to prevent the contact of the gas with any organic substance, indiarubber, cork, &c. All the operations comprising an estimation—the measurement of the volume of the gas, the decomposition of the ozone, the titration with iodine—are all carried out in the apparatus itself.

The apparatus consists of a conical flask of 600 cubic centimetres capacity, closed by means of a hollow ground-glass stopper. This stopper carries a graduated funnel tube, reaching almost to the bottom of the flask, and with a glass tap, A, a little below the funnel; below this tap a side tube is sealed in, serving to introduce the gas; it is also fitted with a tap, B. This tube is fixed in such a position that the liquid running from the funnel cannot enter it. The neck of the flask also carries a side tube sealed in, and the hollow stopper is pierced with a hole, in such a manner that, when this hole is placed opposite a tube, C, the apparatus communicates with the outer air;

knoes of glass sealed on to the stopper serve to facilitate the rotation of the latter in its seat. The capacity of the flask is determined accurately by the weight of water it contains. This capacity is marked on the outside. All the ground parts should form perfect joints, even when dry. If this is not the case, concentrated sulphuric acid must be used as a lubricant, to the exclusion of all fatty substances.

We commence by connecting the apparatus with the source of ozonised gas, avoiding contact of the gas with any organic substance. For this purpose we must use either a ground-glass joint or the hydraulic sealing arrangement devised by Engler and Masse (*Ann. de Chem. et de Phys.*, vol. xciv., p. 225); or again, two brass tubes cemented on to the two glass tubes to be joined, and these are screwed together, making the joint tight with asbestos paper.

If the two tubes to be connected are of the same outer diameter, they may be covered with a piece of glass tube about 2 centimetres in length, of the nearest possible size. The extremities of the two tubes are placed in contact and the covering tube luted on by means of melted paraffin. In this manner we obtain a very good joint, easy to disconnect.

The gas to be examined is passed into the apparatus in such a manner as to completely displace the air it contains. The gas enters through the tube sealed into the funnel stem, and leaves by the lateral tube sealed into the neck of the flask; the taps B and C are open, while A is closed. Once the air is driven out, the tap B is first closed, and then C by a slight rotation of the stopper. If the filling of the apparatus is effected by means of aspiration, it is necessary to proceed in the inverse order. The funnel is filled with a N/5 solution of iodide of potassium, up to one of the graduated marks, and the funnel tap is opened. To make the liquid penetrate into the apparatus, it suffices to place the latter in communication with the outer air for a few instants; this is done by means of the tap C. During this operation a volume of gas is removed exactly equal to the volume of the reagent used, and this must be allowed for in the calculation of results. The absorption of the ozone is almost instantaneous; it gives rise to the formation of white fumes, and even to fumes of iodine, if the proportion of iodine is high; these fumes are rapidly absorbed by frequent agitation. As soon as they have disappeared the stopper is removed and the tubes rinsed, both inside and out, and a quantity of N/10 sulphuric acid added, equal to the amount of iodide used. The iodine set free is titrated with centinormal hyposulphite of soda in the presence of starch. The use of a more concentrated solution of hyposulphite is not recommended because of the instability of such solutions. If the gas under examination contains too great a proportion of ozone, only a known fraction of the iodide liquor is titrated.

It is customary to express the amount of ozone present in grammes per cubic metre of gas. If it is desired to express it in volumes per cent, we take a titrated solution of such a strength that 1 cubic centimetre corresponds to 1 cubic centimetre of supposed gaseous ozone in the normal state. Before making any calculations we must determine the volume of gas on which we have operated, which is done by subtracting from the capacity of the apparatus the volume of the solution of iodide used.

Two pieces of apparatus of the same kind, placed in series and filled with the same gaseous mixture, give perfectly concordant results.

It is just the same if we slowly displace the gas contained in an apparatus by introducing water, and let the gas bubble through two Drechsel washing flasks containing iodide of potassium. From this experiment we may conclude that the time during which the gas is in contact with the iodine is without influence on the completion of the reaction, a result which agrees with that found by K. Garzoroli-Turnlack (*Sitzungsber. d. K. K. Akad. d. Wissensch. Wien*, 1901, cx., II.). It does not matter, as far as the final result is concerned, whether the ozone is

decomposed slowly, by passing it bubble by bubble through a solution of iodide, or whether the whole of the gas is put suddenly in contact with the reagent.

As for the action of the ozone on acidulated solutions of iodide, Ladenburg represents it by the following equation:  $4O_3 + 10HI + H_2O = 5I_2 + H_2O_2 + 5H_2O + 3O_2$ .

The formation of peroxide of hydrogen explains the reappearance of the blue colour in the liquid after the titration by hyposulphite.

I have examined the influence of the time of contact, and of the concentration of the hydriodic acid on the reaction. The duration of the time of contact is without any influence, and the concordance of the results obtained with a given acid solution is as close as that obtained when using a neutral solution. But if we use more and more concentrated solutions of hydriodic acid, we find that the amounts of iodine set free increase for a given ozonised gas. With solutions comprised between N/10 and N/5 we obtain 3 atoms of free iodine per molecule of ozone; with a normal solution we obtain 3.7 atoms, and with a double normal solution, 4.2 atoms.

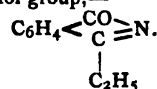
The reappearance of the blue colour in the liquid, after the titration with hyposulphite, which always takes place when the ozone has been passed slowly through a neutral solution of iodine, has never been observed in these experiments. The equation proposed by Ladenburg is therefore at fault in this respect. I have endeavoured to account for the excess of iodine set free in this case by admitting that the ozone exercises a catalytic action on the reaction between the oxygen and the hydriodic acid. This explanation is not plausible, for the excess of iodine set free in a solution of hydriodic acid of given strength, is proportional to the quantity of ozone entering into the reaction, which is contrary to the definition of catalysis.

To explain this particular behaviour, we may make the following supposition. The molecule of ozone decomposes on contact with certain substances, into an atom of very active oxygen and a molecule of inactive oxygen. But this latter possesses, at the moment of its formation, a more powerful oxidising action than that of ordinary oxygen, and it is capable of oxidising certain easily oxidisable bodies, such as hydriodic acid, while it is not able to oxidise other bodies, such as iodide of potassium, ferrous sulphate, or arsenious acid. At the moment when the two atoms do not form a completely closed molecule, we have to deal with a special case of the nascent state.

This supposition is confirmed by the following fact:—When a given quantity of ozonised oxygen is rapidly put in contact with hydriodic acid, the oxidation takes place instantly, and the separation of the iodine, which eventually occurs, is very slight and of the same order as that brought about by contact with pure oxygen.—*Zeitschrift für Angewandte Chemie*, 1903, p. 894.

#### Action of Mixed Organo-magnesium Compounds on Phthalimide and Phenylphthalimide.—Constantin

Béis.—The author investigates the action of  $Mg < \begin{smallmatrix} Br \\ C_2H_5 \end{smallmatrix}$  on phthalimide and phenylphthalimide, and finds that, with the first of these substances, a body is produced which belongs to the isoindol group,—



The Hydrates of Methyl Alcohol and Acetone.—E. Varenne and L. Godefroy.—With methyl alcohol the authors prepare the six hydrates  $CH_3OH + H_2O$ ,  $CH_3OH + 2H_2O$ ,  $CH_3OH + 3H_2O$ ,  $CH_3OH + 4H_2O$ ,  $CH_3OH + 5H_2O$ ,  $CH_3OH + 8H_2O$ ,  $CH_3OH + 20H_2O$ , and they also find that acetone gives with water at least three definite hydrates,  $CH_3-CO-CH_3 + 3H_2O$ ,  $CH_3-CO-CH_3 + 4H_2O$ ,  $CH_3-CO-CH_3 + 8H_2O$ , and probably— $CH_3-CO-CH_3 + 34H_2O$ .

—*Comptes Rendus*, cxxxviii., No. 16.

## PROCEEDINGS OF SOCIETIES.

### THE ROYAL SOCIETY. CONVERSAZIONE AT BURLINGTON HOUSE.

On Friday evening last, May 13, 1904, a *Conversazione* was held at the Society's rooms, Burlington House, Piccadilly. The guests, who numbered about 900, were received by the President, Sir William Huggins, K.C.B., O.M., and the members of the Council.

The exhibits were of an interesting character, though there was nothing of a strikingly novel kind such as the Fellows have become accustomed to expect at these functions. Among those which attracted attention were some excellent examples of Microphotography shown by Mr. ARTHUR SMITH and Mr. RICHARD KERR, F.G.S. Their exhibit included sections of histological, botanical, and entomological specimens, intended to assist students of biology generally and medical students especially. The camera used is unusually large in order to ensure direct photography. In no case were the results produced from the enlargement of small negatives.

Some models and photographs of large Hail-stones were shown by the Royal Meteorological Society; some of these fell in Yorkshire, Sussex, and at Montreux in France, and though rare in Europe they are by no means such curiosities in tropical regions.

Mr. J. W. GORDON's exhibit of High Power Microscopy was of interest. The apparatus exhibited consisted of a compounding draw-tube and oscillating screen, as proposed in J. W. Gordon's paper on the Helmholtz theory of the microscope recently read before the Royal Microscopical Society. The object is to get rid of the blemishes in very highly super-amplified images which Helmholtz proved to be due to the extremely small diameter of the Ramsden circle incidental to excessive magnifying power, and it is accomplished by employing an opalescent screen in the view plane of the principal microscope to serve as a secondary source of radiation, and expand the transmitted wave-front. The screen is kept oscillating in order to render its grain invisible. The object exhibited was a diatom (*Pleurosigma angulatum*) magnified about 10,000 diameters.

Prof. J. A. FLEMING, F.R.S., showed an Apparatus for the metrical study of Stationary Electric Waves on Spiral Wires. The apparatus exhibited consists of a long solenoid of silk covered wire having 5000 turns and a total length of 643 metres. This solenoid has parallel to it an adjustable earth wire and a divided scale. The solenoid is connected to one point on an oscillatory electric circuit consisting of a couple of Leydens having a capacity of 0.00068 mfd. and an adjustable inductance of 0 to 230 microhenrys, and a silent discharger. When oscillations are set up in this circuit by induction coil discharges and the frequency adjusted, stationary electric waves are set up in the solenoid. The position of the loops and nodes is ascertained by the use of a series of carbonic dioxide vacuum tubes.

The exhibit of Dr. A. E. WRIGHT was of special interest; it consisted of apparatus and methods employed for measuring, in the case of human blood, its content in agglutinating substances, bactericidal substances, red blood corpuscles, albuminous substances, calcium salts, and salts generally. Of the methods which were exhibited, some are employed in connection with the study of the changes produced in the blood by anti-typhoid, anti-staphylococcus, and anti-tubercle inoculation; others are employed in connection with the determination of the efficiency of the kidney, and in connection with the study of the conditions of blood which are associated with hæmorrhage, serous effusion, and intravascular coagulation.

Dr. A. B. GREEN showed some photographs illustrative of induced Radio-activity of Bacteria; and Mr. H. JACKSON

exhibited some new Phosphorescent Materials illustrating various degrees of response to such exciting influences as violet and ultra-violet light, electric discharge, heat, and friction.

The Cambridge Scientific Instrument Company exhibited a Vibrograph for Recording Vibrations photographically; and a Micro-manometer designed by Mr. R. THRELFALL, F.R.S., for use in conjunction with a Pitot tube and side gauge, to indicate the velocity of gases in pipes or chimnies.

Mr. H. H. CUNYNGHAME, C.B., showed his Improved Muffle and Melting Furnaces at work. The plan on which these furnaces are constructed is to jacket them thickly with non-conducting material, in such a way that heat cannot escape as fast as it is developed, until a high temperature has been attained.

By this plan, muffle furnaces can be heated with from  $\frac{1}{2}$  to  $\frac{3}{4}$  the amount of gas or petroleum which is required without jacketing, and at the same time evenness of temperature is obtained, and the operator not exposed to heat. The furnaces can be used with petroleum or gas, and as the gas jet for muffles up to 7 inches width is only a single Bunsen burner, no flues are required to carry off the fumes.

During the evening three Demonstrations, with the help of the electric lantern, were given in the Meeting Room. They were by Prof. W. A. HERDMAN, F.R.S., on the Recent Investigation of the Ceylon Pearl Fisheries; Mr. FRANCIS FOX, M.Inst.C.E., who showed Lantern Slides, illustrative of—(1) Operations at the Simplon Tunnel, (2) the Victoria Falls and Gorge of the River Zambesi, and proposed Bridge; and the Hon. C. A. PARSONS, F.R.S., who gave a Demonstration of the Auxetophone, which is an air-operated valve used for a reproducer in gramophones and phonographs, and which replaces the usual reproducing diaphragm in such machines, and the application of this valve to the violin was shown. Selections of music, vocal and instrumental, were played on the auxetophone. The augmentation of the sound by the application of this device was very marked, the increase being from five to eight times that of the ordinary gramophone; there was also a greater fullness of the voice apparent, though in some cases the noise was almost overpowering.

### CHEMICAL SOCIETY. Ordinary Meeting, May 5th, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

MESSRS. C. M. Beadnell, C. T. Bennett, and T. U. Walton were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. W. Eddington Field, 65, Sutherland Road, Armadale, Melbourne, Victoria, Australia; John Hulme, 3, Albert Terrace, Stockport.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—William Barbour, M.A., B.Sc.; R. H. Durward Benn; Robert Currie Bridgett, M.A., B.Sc.; Ellis Clayton; Robert Steel Finlow, B.Sc.; Harold Sankey Hammond; George Mathieson Horn; Percy Kent Le May; Alfred Mander; Tom Sidney Moore, B.A., B.Sc.; Gerald Oscar Morgan Smith; Laurel Cecil Francis Oldfield; William Davidge Page; Gerald Pinchbeck; Thos. Linton Daniel Porter, B.Sc.; Louis John Ezekiel Riley; Franklin Ernest Robertson; Thomas George Shacklady; Clarence Smith, D.Sc.; Henry Heron Smith; Reginald Harry H. Stanger; Robert de J. Fleming-Struthers, B.A.; Walter Tong.

The PRESIDENT announced that the following persons were proposed by the Council for election as Foreign Members:—Henri Becquerel, Cornelis Lobry de Bruyn, Frank Wigglesworth Clarke, Madame Marie Curie, Carl Liebermann, Edward Williams Morley; and stated that, in accordance with By-law II., the ballot for their election would take place at the next meeting of the Society on Wednesday, May 18th.

The PRESIDENT announced that the following letter had been received from Sir Henry Roscoe in reply to the address of congratulation from the Society on the occasion of the celebration of the Jubilee of his Doctorate.

"To the Council and Fellows of the Chemical Society I offer my hearty thanks for the honour they have done me through the President, Professor Tilden.

'Instituted in 1841 for the purpose of stimulating chemical research in this country by the leaders of our science of that day, amongst whom Thomas Graham stands pre-eminent, the influence which the Society has exerted during more than half a century on the progress of English Chemistry has been far greater than its founders could have foreseen.

'During the last fifty years our Science has undergone not one but many revolutions, each one widening and deepening its foundations.

'In this wonderful series of changes, thanks to the zeal and ability of its Fellows, both young and old, the Chemical Society has borne no inconsiderable part, and for its still wider influence and its still greater activity no one has warmer wishes than myself.

(Signed) HENRY E. ROSCOE."

Of the following papers, those marked \* were read:—

\*72. "The Slow Combustion of Ethane." By WILLIAM ARTHUR BONE and WILLIAM ERNEST STOCKINGS.

The authors have studied (1) the interaction of ethane and oxygen at 250–400°, under pressures of 1.75 to 2.33 atmospheres, in borosilicate glass bulbs (compare *Trans.*, 1902, lxxxi., 536); (2) the oxidation of ethane in contact with a surface of porous porcelain at 400–500°, under reduced pressure, in the circulation apparatus described in a previous paper (*Trans.*, 1903, lxxxiii., 1074); and (3) the slow combustion of ethyl alcohol and acetaldehyde. The main conclusions deduced from the experiments are as follows:—

1. The proportions of ethane and oxygen most favourable to chemical change are equimolecular (1 : 1), although interaction takes place nearly as rapidly in mixtures containing ethane and oxygen in the ratio 2 : 1. At all temperatures, ethane is oxidised much more rapidly than is methane, other conditions being equal.

2. When ethane is treated with proportions of oxygen insufficient to oxidise the whole to carbon monoxide and steam, there is no preferential combustion either of hydrogen or carbon. In the absence of secondary phenomena, the interaction is always marked by a diminution in the pressure of the cold products without any deposition of carbon or liberation of free hydrogen.

3. The combustion proceeds in several well-defined stages, among which the following have been distinguished:—

(i.). A primary oxidation to acetaldehyde and steam.

(ii.). A further rapid oxidation of the acetaldehyde to formaldehyde, carbon monoxide, and steam.

(iii.). The final oxidation of formaldehyde to carbon monoxide, carbon dioxide, and steam, which may best be considered as due to two simultaneous interactions—i.  $\text{CH}_2\text{O} + \text{O}_2 \rightarrow \text{CO}_2 + \text{H}_2\text{O}$ ; ii.  $2\text{CH}_2\text{O} + \text{O}_2 \rightarrow 2\text{CO} + 2\text{H}_2\text{O}$ .

4. The combustion of the hydrocarbon may be accompanied by the following secondary phenomena:—

(i.). Hydrogen or methane, or both, may appear in the products without any liberation of carbon, as the result of the purely thermal decompositions of formaldehyde and acetaldehyde vapours respectively, the latter giving rise to methane and carbon monoxide, whilst the former yields hydrogen and carbon monoxide, although its decomposition is a more complex process than that of acetaldehyde.

(ii.). Small quantities of ethylene and hydrogen may appear in the products as the result of the thermal decomposition of ethane.

(iii.). If the heat liberated during the initial stages of oxidation is sufficient to raise the temperature of the interacting gases to the ignition-point, an explosion takes place, during which the excess of hydrocarbon, and probably also

of aldehyde vapours, is decomposed, carbon and hydrogen being liberated together with some acetylene and ethylene. There is no liberation of carbon below the ignition-point.

5. Although ethyl alcohol has not been detected among the oxidation products of ethane, its absence may be explained by the fact that it is oxidised far more rapidly than is ethane under similar conditions.

6. The view taken of the mechanism of the combustion of ethane is supported by the experiments on ethyl alcohol and acetaldehyde.

\*73. "The Action of Radium Rays on the Halides of the Alkali Metals and Analogous Effects produced by Heat." By WILLIAM ACKROYD.

The author has studied the action of  $\gamma$ -rays from radium bromide, in its violet glass tube, on the chlorides of lithium, sodium, potassium, rubidium, and caesium, and deduces the following conclusions:—

1. The colour changes produced divide the compounds into two sub-groups:—I. LiCl, NaCl, and II. KCl, RbCl, CsCl, and in the effects observed: LiCl, white; NaCl, orange; KCl, violet; RbCl, bluish-green; and CsCl, green, each sub-group conforms to the constitutive-colour law (*CHEMICAL NEWS*, 1893, lxxvii., 27).

2. The division into these sub-groups is also indicated by their differences of molecular aggregation as expressed by 1/sp. vol.

3. There is relative stability of the colours produced while they remain in darkness, and their rate of disappearance or decay in day-light varies with the intensity of the light.

4. The relative expenditure of energy in the  $\gamma$ -rays effecting the colour changes decreases with increase of molecular weight, or, in other words, the sensibility to the action of the  $\gamma$ -rays increases with the molecular weight.

5. When the induced phosphorescence has decayed so as to be no longer visible, it can be revived by invisible heat. In the case of fused lithium chloride, a temperature of 37° is sufficient for the purpose.

6. These various features correspond in many respects with the thermal effects produced in other substances, and the whole of the evidence points to the conclusion that both are physical changes.

\*74. "The Mutarotation of Glucose and of Galactose. Solubility as a means of Determining the Proportions of Dynamic Isomerides in Equilibrium." By THOMAS MARTIN LOWRY.

The method formerly described (*Proc.*, 1903, xix., 156), and applied to the case of  $\beta$ -bromonitrocampbor, has now been used in order to determine the proportion of  $\alpha$ -glucose and of  $\alpha$ -galactose in the mixtures of dynamic isomerides which are formed when these sugars are dissolved in alcohol or in water. The solubility of  $\alpha$ -glucose in anhydrous methyl alcohol gradually increases in the course of a week in the ratio of 0.53 : 1, and that of  $\alpha$ -galactose in the ratio 0.50 : 1. The increment of solubility is due to the formation in solution of the stereoisomeric  $\beta$ -glucose (Tanret's  $\gamma$ -glucose) and  $\beta$ -galactose; when equilibrium is reached, the quantity of sugar in solution is nearly twice as great as when the  $\alpha$ -form alone is present, and the stereoisomerides must therefore be in approximately equal proportion and be almost equally stable. This result differs from that obtained by Jungius in the case of the methyl-glucosides, for which the ratio was found to be  $\alpha : \beta = 3 : 1$  instead of 1 : 1.

In a mixture having approximately the composition  $\text{EtOH} + \text{H}_2\text{O}$ , the solubility of  $\alpha$ -galactose was found to increase in the ratio 0.39 : 1 and in a mixture having approximately the composition  $\text{PrOH} + 2\text{H}_2\text{O}$  in the ratio 0.35 : 1. The decrease of the ratio in the aqueous-alcoholic solutions may be attributed to the presence in the solution, along with the stereoisomeric  $\alpha$ - and  $\beta$ -galactoses, of a considerable proportion of the hydrated aldehydic form of the sugar, which is probably an intermediate product in the isomeric change (compare *Trans.*, 1903, lxxxiii., 1316); this conclusion is in harmony with the fact that the rate of

change is many times greater than in the anhydrous methyl-alcoholic solutions.

Similar experiments were made on the dissolution of  $\alpha$ -glucose in mixtures having approximately the composition  $2\text{EtOH} + \text{H}_2\text{O}$  and  $\text{EtOH} + \text{H}_2\text{O}$ , but in each case the gradual increase of solubility was preceded by a more or less rapid decrease owing to the conversion of the solid  $\alpha$ -glucose into a less soluble hydrate.

\*75. "A Study of the Substitution Products of *ar*-Tetrahydro- $\alpha$ -naphthylamine. 4-Bromo-*ar*-tetrahydro- $\alpha$ -naphthylamine and *ar*-Tetrahydro- $\alpha$ -naphthylamine-4-sulphonic Acid." By GILBERT THOMAS MORGAN, FRANCES MARY GORE MICKLETHWAIT, and HERBERT BEN WINFIELD.

The acyl derivatives of *ar*-tetrahydro- $\alpha$ -naphthylamine (Bamberger and Althaus, *Ber.*, 1888, xxi., 1895), when brominated with one molecular proportion of the halogen or hypobromous acid, ultimately undergo substitution in the aromatic ring, the entrant bromine atom assuming the para-position with respect to the aminic nitrogen. The bromo-base obtained on hydrolysis was shown to be 4-bromo-*ar*-tetrahydro- $\alpha$ -naphthylamine (m. p.  $42^\circ$ ) by furnishing successively 1-bromotetrahydronaphthalene and 1-bromodinitrotetrahydronaphthalene (m. p.  $91^\circ$ ). The isomeric 2-bromodinitrotetrahydronaphthalene prepared from *ar*-tetrahydro- $\beta$ -naphthylamine for the purpose of comparison melted at  $105$ – $106^\circ$ . On treatment with diazo-compounds, the bromo-base yields diazoamines of the type  $\text{C}_{10}\text{H}_{10}\text{Br}\cdot\text{NH}\cdot\text{N}_2\text{X}$ . For example, 4 : 4'-dibromo-1-diazo-1-aminotetrahydronaphthalene prepared by the interaction of the base with its own diazonium salt decomposes violently at  $190$ – $194^\circ$ , and when treated with cold concentrated hydrochloric acid undergoes fission, yielding the hydrochlorides of its generators, the diazonium chloride being identified by the formation of 4-bromotetrahydronaphthalene-*azo*- $\beta$ -naphthol (m. p.  $215^\circ$ ).

4-Bromo- $\alpha$ -naphthylamine, unlike the analogously substituted tetrahydro-base, condenses with diazo-compounds to form ortho-azo-derivatives having the general formula  $\text{XN}_2\cdot\text{C}_{10}\text{H}_7\text{Br}\cdot\text{NH}_2$ . The *azo*-compound,—



yields, on reduction, sulphanilic acid and naphthylene-1 : 2-diamine.

*ar*-Tetrahydro- $\alpha$ -naphthylamine-4-sulphonic acid,  $\text{NH}_2\cdot\text{C}_{10}\text{H}_{10}\cdot\text{SO}_3\text{H}\cdot\text{H}_2\text{O}$ , produced by sulphonating *ar*-tetrahydro- $\alpha$ -naphthylamine, was converted into tetrahydronaphthalene-1-sulphonic acid by eliminating its amino-group, the same product being also obtained by replacing this radicle in *ar*-tetrahydro- $\alpha$ -naphthylamine by the  $\text{SO}_2\text{H}$  group and oxidising the tetrahydronaphthalene-1-sulphonic acid thus produced. Tetrahydronaphthalene-1-sulphonic chloride and the corresponding anilide melt at  $70.5^\circ$  and  $144$ – $145^\circ$  respectively.

*ar*-Tetrahydro- $\alpha$ -naphthylamine-4-sulphonic acid differs from  $\alpha$ -naphthylamine-4-sulphonic (naphthionic) acid in yielding diazoamino-derivatives and not azo-compounds.

The azo-derivatives of *ar*-tetrahydro- $\alpha$ -naphthylamine were shown to be para-azo-compounds like the corresponding azo- $\alpha$ -naphthylamines, for, on reduction, they gave rise to *ar*-tetrahydronaphthylene-1 : 4-diamine.

These results indicate that although *ar*-tetrahydro- $\alpha$ -naphthylamine resembles  $\alpha$ -naphthylamine in forming para-azo-compounds with greater facility than a benzenoid amine, yet its para-substituted derivatives, unlike those of  $\alpha$ -naphthylamine, do not yield ortho-azo-compounds, and in this respect behave like *p*-bromoaniline and other similarly substituted bases of the benzene series.

\*76. "Studies in the Tetrahydronaphthalene Series. Part II. Halogen Derivatives of *ar*-Tetrahydro- $\beta$ -naphthylamine." By CLARENCE SMITH.

In a previous paper (*Trans.*, 1902, lxxxi., 900), it was shown that *ar*-tetrahydro- $\beta$ -naphthylamine resembles primary benzenoid amines in giving diazoamino-com-

pounds, and this resemblance is exemplified further in its behaviour towards the halogens.

Aceto- $\beta$ -naphthalide yields only 1-bromoaceto- $\beta$ -naphthalide at the ordinary temperature, even with excess of bromine. *ar*-Tetrahydroaceto- $\beta$ -naphthalide reacts with one molecular proportion of bromine to form two compounds, namely, 1-bromo-*ar*-tetrahydroaceto- $\beta$ -naphthalide and 4-bromo-*ar*-tetrahydroaceto- $\beta$ -naphthalide.

The following compounds have been prepared :—

1-Bromotetrahydronaphthalene,  $\text{C}_{10}\text{H}_{11}\text{Br}$ , obtained from *ar*-tetrahydro- $\alpha$ -naphthylamine by the Sandmeyer reaction, is a colourless, refractive liquid with an odour resembling that of halogen compounds of the benzene series; it is volatile in steam, and dissolves readily in organic solvents (b. p.  $255$ – $257^\circ/751$  m.m.).

2-Bromotetrahydronaphthalene, prepared from *ar*-tetrahydro- $\beta$ -naphthylamine, resembles the preceding compound, and boils at  $238$ – $239^\circ$  under 758 m.m. pressure.

1-Bromo-*ar*-tetrahydroaceto- $\beta$ -naphthalide,  $\text{C}_{12}\text{H}_{14}\text{ONBr}$ , resulting from the action of one molecular proportion of bromine on an equivalent quantity of *ar*-tetrahydroaceto- $\beta$ -naphthalide dissolved in glacial acetic acid, separates from alcohol in colourless, octahedral crystals (m. p.  $125.5^\circ$ ).

4-Bromo-*ar*-tetrahydroaceto- $\beta$ -naphthalide, obtained from the mother-liquor in the preparation of the preceding isomeric 1-bromo-compound, separates from alcohol in colourless needles (m. p.  $151^\circ$ ).

1-Bromo-*ar*-tetrahydro- $\beta$ -naphthylamine,  $\text{C}_{10}\text{H}_{12}\text{NBr}$ , is prepared by hydrolysing the corresponding acetyl derivative; it separates from light petroleum in colourless, silky needles (m. p.  $52.5^\circ$ ). It dissolves easily in alcohol or acetone, and is volatile in steam.

4-Bromo-*ar*-tetrahydro- $\beta$ -naphthylamine crystallises from light petroleum in colourless needles (m. p.  $52^\circ$ ); it is not readily volatile in steam.

\*77. "Studies in the Tetrahydronaphthalene Series. Part III. Reaction between *ar*-Tetrahydro- $\beta$ -Naphthylamine and Formaldehyde." By CLARENCE SMITH.

Evidence of the benzenoid nature of *ar*-tetrahydro- $\beta$ -naphthylamine is obtained from the study of the behaviour of the base towards formaldehyde. The product is methylene-*ar*-tetrahydro- $\beta$ -naphthylamine, analogous to methyleneaniline. There is no tendency to produce an acridine, which would be evidence of its naphthalenoid character.

The following compounds have been prepared :—

Methylene-*ar*-tetrahydro- $\beta$ -naphthylamine,  $(\text{C}_{11}\text{H}_{13}\text{N})_3$ , is obtained together with a polymeric modification,  $(\text{C}_{11}\text{H}_{13}\text{N})_x$ , when a solution of formaldehyde is added slowly to *ar*-tetrahydro- $\beta$ -naphthylamine. The trimolecular form separates from acetone or light petroleum as a felted mass of small, white needles; it melts at  $121^\circ$ , and gives an intense blood-red colouration with glacial acetic acid.

The polymeric modification is a white, amorphous powder which, when heated, darkens at  $156^\circ$  and fuses at  $164^\circ$ . It is almost insoluble in acetone, light petroleum, or benzene, but dissolves in hot alcohol, and becomes converted mainly into the trimolecular form.

Methyl-*ar*-tetrahydro- $\beta$ -naphthylamine,  $\text{C}_{10}\text{H}_{11}\cdot\text{NH}\cdot\text{CH}_3$ , obtained by the reduction of methylene-*ar*-tetrahydro- $\beta$ -naphthylamine by sodium and amyl alcohol, is a colourless, refractive, oily liquid which boils at  $267.5^\circ$  under 210 m.m. pressure. The hydrochloride,  $\text{C}_{11}\text{H}_{13}\text{N}\cdot\text{HCl}$ , crystallises from water in white needles. The nitrate,  $\text{C}_{10}\text{H}_{13}\text{N}\cdot\text{HNO}_3$ , separates in needles when an ethereal solution of the base is shaken with dilute nitric acid. The nitrosoamine,  $\text{C}_{10}\text{H}_{11}\text{N}(\text{CH}_3)\cdot\text{NO}$ , is a yellow oil, which gives Liebermann's reaction.

\*78. "Note on the Hydrolysis of Starch by Diastase." By JOHN SIMPSON FORD.

The writer finds that Kjeldahl's "law of proportionality" is true for the diastase of barley and air-dried malt, as well as for that of kiln-dried malt.

(To be continued.)



## PHYSICAL SOCIETY.

Ordinary Meeting, May 6th, 1904.

Mr. J. SWINBURNE, Vice-President, in the Chair.

A PAPER entitled "*Some Instruments for the Measurement of Large and Small Alternating Currents*" was read by Mr. W. DUDDELL.

The author, after some preliminary remarks on the available means for measuring alternating currents, proceeded to describe three thermal instruments which he has constructed for this purpose. The first instrument is essentially a sensitive Ayrton-Perry twisted strip ammeter which is very quick in action for a thermal instrument, and has been used for observing and recording P.D.'s and currents which varied as rapidly as one per second. It is compensated for change in the surrounding temperature by forming the sides of the frame which holds the twisted strip with the same wire that the strip itself is made from. With the instrument exhibited a current of 22 milliamperes gave a deflection of one-quarter the scale-distance, i.e., 250 m.m. at one metre scale-distance. The mechanical periodic time is only about one-fifteenth of a second. Using this instrument in series with a high resistance the author has made observations on the variations in the voltages of alternators caused by cyclic irregularity of the engine. By working to a false zero it is easy to obtain 10 m.m. change in deflection for 1 per cent change in the P.D. The second instrument exhibited was a very sensitive thermal galvanometer called in the paper a "Thermogalvanometer." It consists of the combination of a radio-micrometer of the "Boys" type with a very small resistance which is heated by the current to be measured, and which in turn heats the thermojunction of the radio-micrometer by radiation and convection. The principle of its action is as follows:—A loop of wire has its two ends fixed to the two bars of a single thermojunction, a mirror is fixed to the loop, and the whole is suspended in a magnetic field by means of a quartz fibre. The heat from the resistance raises the temperature of the thermojunction, and causes a current to flow round the loop which is deflected by the magnetic field. The sensibility of the instrument depends on the resistance of the heater. Using a heater having a resistance of 13,970 ohms, a deflection of 250 m.m. at a scale-distance of one metre is obtained with a current of 31 microamperes; a heater having 18 ohms resistance required 800 microamperes to give the same deflection. To illustrate the high sensibility of this instrument the author showed the large deflections produced by the currents through a telephone receiver even when the source of sound was many feet distant from the microphone; he also showed that if the thermogalvanometer was placed in series with the vertical receiving wire in spark telegraphy over a short distance, large deflections were produced. The third instrument described was a switchboard instrument which works on the same principle as the last, only that the moving part is pivoted in the usual way. The author exhibited one of these instruments, arranged to give the whole scale deflection for only 0.15 volt, which can be used in connection with shunts to measure large currents; for instance, to measure 1000 amperes the power lost in the shunt would only be 150 watts. Transformers can also be used, as the power to produce the whole scale-deflection is only 0.3 watt. A similar instrument with a high-resistance heater was also exhibited giving the whole scale-deflection for 0.1 ampere, which can be used as a voltmeter by putting resistance in series with it.

Prof. R. THRELFALL, in a letter to the Secretary, congratulated the author on his instruments, and stated that we were not told the effect of change of relative position in the heater and thermojunction during the operation of the thermogalvanometer. This effect might make it possible to construct an instrument having either a scale of equal parts or of any desired form.

Dr. P. E. SHAW, in a letter to the Secretary, said it was

possible to measure the amplitude of vibration in the diaphragm of a telephone-receiver, and Mr. Duddell's instruments would enable us to determine the currents producing these vibrations.

Dr. D. K. MORRIS pointed out that the working forces in an electrostatic instrument could be increased by limiting the angular range without increasing the moment of inertia of the moving system. For instance, this could be effected in a quadrant electrometer with a wide needle by subdividing the needle into narrow fingers, and subdividing the quadrants in a similar manner. Dr. Morris showed an instrument constructed on this principle capable of measuring 0.1 volt when used idiostatically. The limit to this process of sub-division is decided by the practical working clearance; the clearance being determined by the tilting of the needle due to unbalanced electrostatic forces. Such an instrument may be called a *multipolar electrostatic instrument*, and Dr. Morris has used the same idea in the construction of a *multipolar electrodynamic instrument*. Both of these instruments were exhibited at the meeting. Dr. Morris said he was glad of the opportunity of Mr. Duddell's paper for bringing them forward, and although they were well suited for use as zero instruments as detectors, they were not nearly so sensitive as the instruments designed by the author of the paper.

Prof. W. E. AYRTON said we had another example of the ingenuity of Mr. Duddell, and drew attention to the fact that he had not only designed the instruments but had also constructed them himself. He was pleased to see that good results had been obtained by using an Ayrton-Perry twisted strip. The use of such strips had hitherto been limited by the absence of contrivances for compensating temperature-change effects. With such compensation it is possible by using a twisted strip to combine simplicity of construction with constancy of zero. In expressing especial interest in the experiment on spark telegraphy, Prof. Ayrton referred to the fact that Mr. Duddell had designed his thermogalvanometer for use in an important investigation, being unable to purchase a suitable instrument. The author had spoken about an alternator with a frequency of 120,000, and he hoped we would soon have some account of it.

Referring to the remarks of Dr. Morris, he said it was often difficult to calculate the sensibility of a quadrant electrometer with a divided needle. In order to do so it was essential that the edges of the needles should never be near to the edges of the inductors. Any arrangement to increase the sensitiveness of an electrostatic instrument was desirable, and if Dr. Morris had obtained large deflecting forces without bringing the needles and inductors very near together, he was to be congratulated. He asked the author if it would not be better in his thermogalvanometer to double the heater on itself, and thus obtain a greater heating effect with diminished self-induction.

Mr. A. CAMPBELL expressed his interest in the instruments shown. Some years ago, in connection with the measurement of small alternating magnetic fluxes, he used a hot wire near a minute thermopile connected with an ordinary moving-coil galvanometer. One of the main difficulties with such arrangements was the slowness in reaching a steady deflection and in settling back to zero. For some relative positions of the thermopile and heater the deflection passes through its steady value, and creeps back to it; for other positions the steady value is reached asymptotically. Even in Mr. Duddell's instruments the effect is quite noticeable, and it appears to want further elucidation. In order to render more uniform the calibration of the scale (which normally follows the square law), Mr. Campbell had sometimes mounted the heater so that by its expansion (or that of another hot wire) a spring drew it further from the thermopile as the temperature of the heater rose.

Mr. K. EDGUMBE congratulated the author, and said that as far as small alternating currents were concerned the instruments shown would be very useful. He could not, however, follow the author when he said that his

pivoted form of thermogalvanometer would make a good switch-board instrument, because it was never necessary to measure small currents. In using Mr. Duddell's instruments it was necessary to have very good contacts. Mr. Duddell had spoken of the advantage of being able to remove one heater and put in another, but the speaker pointed out that it would be necessary to adjust the second heater with accuracy, or errors would be introduced. He was not a believer in hot-wire instruments, which were unsuitable for central-station work, and gradually dying out in this country.

Mr. W. A. PRICE said he had recently been thinking of the possibilities of the instrument described by Dr. Watson in the discussion on Prof. Fleming's recent paper on a "Hot-wire Ammeter." He thought that in many respects this instrument would be more practical than those brought forward, although it would probably not be so sensitive.

Mr. W. BENNETT asked how far the temperature correction in the twisted strip was perfect. It might be slightly affected by the fact that the twisted strip, though of the same material as the side wires, had been treated rather roughly in the process of flattening and twisting, the coefficient of expansion being possibly lessened. The fact that the strip and wires were at different temperatures might also affect the correction adversely on account of the temperature variation of elasticity. The sensitiveness of the radio-micrometer form of instrument would be increased if the efficiency of heat-transference between the heater and the thermojunction could be improved. If it were possible to make a heater that would nearly surround the junction this might be effected, and the variation of sensitiveness due to inaccurate replacement of the heater after removal would be lessened. He suggested using as a heater a thin film of platinum deposited on the inside of a small thin tube of glass or quartz.

Mr. R. S. WHIPPLE said he would like to testify to the great patience of Mr. Duddell in overcoming the difficulties met with in the construction of his instruments. He was of the opinion that a good deal of the bad name of hot-wire instruments could be attributed to their inability to stand overload.

Mr. H. TINSLEY, referring to Prof. Ayrtton's remarks about the position of the needle in an electrostatic instrument, said he had recently tested the matter accurately. So long as the edges of the needle were well within the inductors it was easily possible to calculate the deflection which would be produced by any voltage to about one part in five hundred. Under such conditions the instrument accurately followed a square law, and its working-constant could be obtained from one observation.

The CHAIRMAN said that a thermopile had been used many years ago in a similar way to that in which the thermojunction had been used by Mr. Duddell, but the sensitiveness obtained was not nearly so great as that of the instruments exhibited. With regard to electrostatic instruments he did not see any advantage in being able to predict the deflection produced by any voltage, according to a definite law, as it was always easy to calibrate such instruments. The shape of an instrument with an Ayrtton-Perry twisted strip often rendered it unsuitable for many purposes.

Mr. W. DUDELL, in reply, said that it was necessary that the heater and the thermojunction should be very small, and this made it difficult to diminish the self-induction of the heater by doubling it on itself. It was also essential that the bars of the thermojunction should be narrow, otherwise creeping effects were introduced. The deflection might also be too small, due to the increase in resistance of the whole circuit brought about by the heating effect of the current. Referring to Mr. Edgcombe's remarks, he pointed out that his pivoted instrument used less watts than any instrument on the market, and could be used if necessary in connection with transformer instruments.

Mr. F. E. SMITH exhibited and described the following instruments from the National Physical Laboratory:—

1. A mercury-resistance standard.
2. A 10-ohm build-up resistance-box.
3. An astatic galvanometer.

An "Experiment with Lubricating Oils," by Mr. W. A. PRICE, was postponed.

## NOTICES OF BOOKS.

*Imperial University of Tokyo; The Calendar 1903-1904.*  
Tokyo: Published by the University. 1904. Pp. 243 + 108.

THIS Calendar contains full information of all kinds concerning the University of Tokyo, commencing with an historical summary and the events leading to the founding of the University. This University is of no great antiquity, for it practically came into existence in March, 1886, though what has been called the "awakening" of Japan was some thirty years earlier.

The course of instruction includes practically every modern subject, and we have had recently, and are still having, striking evidence of the thorough manner in which our friends the Japanese attend to detail; we may therefore be certain that the peaceful arts receive equal care and attention.

*Cyaniding Gold and Silver Ores; a Practical Treatise on the Cyanide Process.* By H. FORBES JULIAN and EDGAR SMART. A.M.I.C.E. With numerous Illustrations and Working Drawings in the Text, and 35 on Folding Plates. London: Charles Griffin and Co., Ltd. 1904. Pp. 405.

THE cyanide process being comparatively new, textbooks, which can be also looked upon as works of reference, are rather rare; much information on the subject has been published from time to time, but it is so scattered among various periodicals all over the world as to be practically inaccessible to the busy man.

In this volume the authors have brought together a great deal of fragmentary information which has appeared from time to time in books, periodicals, and the transactions of learned societies, and have arranged it in such a form as to make a complete story of cyaniding from its earliest beginnings to its latest developments. That they are well qualified for this task is evident to anyone who reads this book. Practice and theory have been made to go hand in hand, so that the mill manager, while finding numerous hints and much detailed information of a practical kind, will be also led to exercise his reasoning powers through studying the principles involved in the working of the process and the construction of the plant.

The book is divided into forty-four chapters, the first four being on preliminary investigations; without going through them *seriatim* we may point out that all the operations involved in the complete working of the process are taken in order. Crushing, measuring, leaching, solution, precipitation and cleaning up, refining and smelting, each have their own section, besides numerous other chapters for the discussion of the side issues involved, such as the effects of temperature, concentration (a very important point), the absorption of air by solutions, sources of loss of cyanide, &c.

The above mentioned chapters deal with the direct extraction of gold from the crushed ores; there is, however, another most important source of gold recovery which was for many years entirely overlooked; we refer to the recovery of gold from slimes.

Naturally, the gold present in slimes is in an extremely finely divided state, and can be extracted only by getting it into solution. The principles and practice of the mani-

pulation involved in this operation are next carefully gone into.

We then come to chapters on the design and construction of the cyaniding plant, the vats, pumps, launders, precipitation boxes, &c., the details of which have to be considered and modified at almost every mine. Finally, the cost of cyaniding is fully considered, both as regards capital outlay and maintenance and cost of treatment.

On all these matters the authors speak with authority, as besides being theoretical and scientific experts, they have the additional and equally great advantage of many years of practical work and close connection with other well-known men in the same field, and can add the knowledge of experience to that acquired by study.

*Radium and all About it.* By S. BOTTONE. London: Whittaker and Co.

THE interest surrounding the discovery and properties of radium is so general that a pamphlet of this character, written for the lay public, is likely to be largely read by those who have neither time nor opportunity to follow the strictly scientific journals. So much nonsense has appeared in print about radium that we welcome a work of this character as a real effort to put the facts concerning one of the greatest discoveries of the century simply and clearly before the public.

Commencing with the observation and properties of cathode rays, and the work of Lenard and Röntgen, the main facts of the discovery and properties of radium and its companions are collected and condensed into small compass. As yet we have no knowledge of the properties of pure radium; all that is known of the element has been gained by a study of exceedingly small quantities of its compounds, chiefly the chloride and bromide; and this fact is kept in mind when discussing, later on, the theoretical considerations involved in the discovery. The note in this connection is worth quoting in full:—"The fact is, that the amount of radium, even in the form of bromide, chloride, or nitrate, at our disposition, has been, up to the present time, so minute that it is premature to build up any theory which, when we can experiment upon pure radium in an uncombined state on a larger scale, may have to be greatly modified, if not altogether discarded. The very fact that not only helium but oxygen, hydrogen, carbon dioxide, and nitrogen are found included in radium bromide, shows how essential it is to the solution of the problem that a fair quantity of pure radium should be experimented upon before any satisfactory theory can be formulated."

Unfortunately, in a compilation of this character, repetitions and the record of erroneous conclusions appear to be unavoidable; and it is a pity that Sir W. Huggins's early statement, that the phosphorescence spectrum of radium bromide gave the helium bands, is quoted, especially as upon this conclusion the author indulges in a good deal of theoretical detail as to the breaking up of the radium atom into that of helium. Later on, the further communication is quoted, showing that the nitrogen and not the helium spectrum is given by the phosphorescence of radium bromide; and that, in so far as this observation is concerned, it gives no support to the alleged transmutation of radium into helium. In this connection, the following quotation from Prof. Rutherford is worth keeping in mind:—"The interpretation of experimental results of such great importance on the transformation theory must naturally be accepted with reserve, until it is proved beyond doubt that the helium present in radium is continuously produced from itself by that element, and cannot be derived from an external source."

On the whole, the main facts about radium are clearly stated, and the book may be commended as an attempt to set out all that is known about one of the most noteworthy victories of patient investigation in such a way that the general reader can form a true idea of the matter.

## CORRESPONDENCE.

### THE CAMPHOR MONOPOLY IN JAPAN.

*To the Editor of the Chemical News.*

SIR,—The Japanese Government, who in the latter portion of last year decided to monopolise the camphor industry of that country, are now well on the way towards reaping a rich harvest as the result of it, and therefore the following remarks may prove interesting:—

The total output of camphor and camphor oil in Japan a few years ago was, roughly speaking, 1,200,000 kin—or, in the English equivalent, 1,600,000 lbs.—but this has increased to such an extent that, in the year 1901, it reached a total of five and a-half millions kin, or 7,333,334 English pounds.

The world's total consumption of camphor alone amounts to five million kin or about seven million pounds per annum, and of this over four-fifths is supplied by Japan and Formosa. The actual amounts supplied by these two countries in the years 1899 to 1902 being as follows:—

|      |       |                                  |
|------|-------|----------------------------------|
| 1899 | .. .. | 2,758,625 kin, or 3,678,134 lbs. |
| 1900 | .. .. | 3,280,815 " " 4,375,420 "        |
| 1901 | .. .. | 4,165,757 " " 5,554,342 "        |
| 1902 | .. .. | 3,953,211 " " 5,270,948 "        |

From these statistics, it will be seen that a monopoly on camphor, which has now been directed by the Government of the Rising Sun, will do much to better the condition of their finances without any actual taxation of the people themselves. It is estimated in official circles in the Orient that, by this monopoly alone, without affecting the price of camphor to any material extent, the Japanese Government will realise annually a sum of 3,000,000 yen, or, in English money, about £300,000 sterling, which will be used to relieve the rates.—I am, &c.,

STANLEY ROE.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 16, April 18, 1904.

**Presence of Argon in the Gases of the Hot Springs of Guadeloupe.**—H. Moissan.—The author examines two specimens of gas from the hot springs of Guadeloupe, and finds that they are volcanic in character, containing completely combusted carbon products, owing to the admixture of air. Fouqué showed the same thing in the gases from eruptions of Santorin. The small quantity of sulphuretted hydrogen which remains in the gas in both cases either escapes combustion by reason of the excess of water vapour in which it is dissolved, or exists as the result of secondary reactions which take place at a high temperature in presence of water vapour.

**Action of Silicon on Water at a Temperature of 100°.**—H. Moissan and F. Siemens.—If pure amorphous or crystalline silicon, reduced to a fine powder, is placed in a little glass tube containing distilled water, and the whole brought to a temperature of 95° and left for some hours, decomposition of the water takes place, with production of silicon hydride.

**Apparent Diminution of Energy of a Dilute Acid in presence of a Neuter Salt of this Acid.**—G. Chesneau.—The production of an alkaline sulphide by the action of H<sub>2</sub>S on sodium acetate, even in presence of free acetic acid, is the cause of the apparent dilution of the latter, and of the non-precipitation in presence of acetic acid, even of the same concentration. It is therefore not

necessary to employ the theory of electrolytic dissociation to explain these phenomena.

**Spectrum of Zinc.**—Maurice Hamy.—The author re-measures certain of the lines in the zinc spectrum, and compares them with the numbers obtained by Perot and Fabry. The agreement is very satisfactory.

|           | Hamy.     | Perot and Fabry. |
|-----------|-----------|------------------|
| 1 .. .. . | 0.6362346 | 0.6362345        |
| 2 .. .. . | 0.5181984 | —                |
| 3 .. .. . | 0.4810533 | 0.4810535        |
| 4 .. .. . | —         | 0.4722164        |
| 5 .. .. . | 0.4680138 | 0.4680138        |
| 6 .. .. . | 0.4629810 | —                |

**Methylic Ether of Acetol,  $H_3C-CO-CH_2(OCH_3)$ .**—Louis Henry.—The author employs the reaction of methylic alcohol on pyruvic formate,  $H_3C-CO-CH_2(CHO_2)$ , to evolve a method for the preparation of the acetol  $H_3C-CO-CH_2(OH)$ , an alcoholic compound which has hitherto only been obtained with great difficulty by the ordinary methods of saponification of the ether salts. This method of preparation is also interesting from a theoretical point of view.

**Methyl Acetolate.**—André Kling.—Methyl acetolate,  $C_3H_5O-O-CH_3$ , can be obtained, not only by the action of dry  $CH_3OH$  on acetol formate, but also by the direct reaction of these two alcohols on one another. One part of acetol and two parts of pure and dry methylic alcohol are heated together at  $140^\circ$  for eight or nine hours, and the crystalline product is then obtained by simple evaporation. When prepared by either method, the product is completely purified by two crystallisations from boiling chloroform. The author further examines its properties and physical constants.

**Halogen Ether Oxides,  $RO(CH_2)_nX$ : their Magnesium Compounds,  $RO(CH_2)_nMgX$ .** New Synthesis in the Tetramethylene Series.—J. Hamonet.—Up to the present time those biprimary halogenated ether oxides,  $RO(CH_2)_nX$ , in which  $n$  equals 1, 2, or 3, have never been prepared. The author succeeds, however, in preparing certain of their superior homologues by a method which may be found capable of generalisation.

**A New Reaction of Aldehydes.**—L. J. Simon and A. Conduché.—The action of acetylacetic ether on aldehydes in presence of ammonia led Hantzsch to evolve a method of synthesis in the pyridic series. More recently, Guareschi undertook parallel researches on the derivatives of glutaric imide. In the same manner, oxalacetic ether condenses with aldehydes in presence of ammonia, giving substitution derivatives of ketopyrrolidone. When benzylic aldehyde is used, a substance with formula  $C_{13}H_{13}NO_4$  is formed. The author examines its properties and prepares its potassium, ammonium, silver, and copper salts.

**Chloruration of Phenyl Carbonate in presence of Antimony Chloride.**—Et. Barral.—For the chloruration of phenyl carbonate the author uses a mixture of anhydrous aluminium chloride and antimony pentachloride. The reaction takes place very slowly in the cold, but more rapidly on heating, the liquid becoming blackish. The chloruration stops after the formation of trichlorophenyl carbonate.

**Action of Sulphur and Selenium on Organo-magnesium Compounds of Mono- and Di-halogen Aromatic Hydrocarbides.**—F. Taboury.—The author examines the action of sulphur and selenium on organo-magnesium compounds of mono-, bromo-, and di-halogen aromatic hydrocarbides, and finds the reaction similar to that investigated by M. Bodroux on the action of oxygen on these compounds. He finds that, at the same time as thio- and seleno-phenols, a more or less quantity of disulphide or diselenide is also produced, proving that the compounds formed during the reaction are oxidised. The author tabulates the compounds formed and their fusing-points.

**Purification and Identification of Alcohols.**—L. Bouveault.—The purification and identification of alcohols,

is rendered more difficult by the fact that these substances are nearly always liquid and have very few crystalline compounds. The author obtains crystalline derivatives of the various alcohols by preparing the pyruvic ethers. These can be easily purified and identified.

**Two Isomeric  $\beta$ -Methylcinnamic Acids.**—M. Tiffeneau.—In a previous paper the author describes the preparation of the two  $\beta$ -methylcinnamic acids by the action of  $CO_2$  on the magnesium derivative of  $\alpha$ -methyl- $\omega$ -bromostyrolene. He succeeds in separating the two acids, owing to their difference of solubility, either in petrol ether or in carbon disulphide. By repeating the crystallisations, he obtains finally two acids of fusing-points  $129^\circ$  and  $97-98^\circ$ . These two acids may be considered as isomers of position—one being phenylcrotonic acid,  $CH_3-C(C_6H_5)=CH-CO_2H$ , and the other a phenyl-isocrotonic acid,  $CH_2=C(C_6H_5)-CH_2-CO_2H$ .

## MISCELLANEOUS.

**Royal Institution.**—On Tuesday next, May 24, at 5, Mr. H. F. Newall begins a course of two lectures at the Royal Institution on the "Solar Corona"; on Thursday, May 26, at the same hour, Mr. H. G. Wells will deliver the first of two lectures on "Literature and the State"; and on Saturday, May 28, at 3 o'clock, Sir Martin Conway begins a course of two lectures on "Spitsbergen in the Seventeenth Century." The Friday Evening Discourse on May 27 will be delivered by the Prince of Monaco, on the "Progress of Oceanography"; and on June 3 by Prof. Svante Arrhenius, on the "Development of the Theory of Electrolytic Dissociation."

**"The Times" Engineering Supplement.**—*The Times* is so big that nobody, in a general way, has time to read it all; still it contains many articles and letters which should, and would, be by no means overlooked if due attention were drawn to them. By the publication of the "Engineering Supplement" the Editor of *The Times* is bringing this subject nearer to the class of readers for whom it is primarily intended, and we have no doubt that this progressive policy will be appreciated, and will meet with the success it deserves. Engineering is the backbone of English prosperity, and every help it receives is bound to have effect by increasing efficiency, and thus benefiting the country at large.

**Silicic Acid (I.).**—E. Jordis and E. H. Kanter.—By means of measurements of conductivity the authors have arrived at the following conclusions:—The existence of the silicates such as  $SiO_3H(NH_4)$  and  $SiO_3(NH_4)_2$  may be considered as certain, that of the silicate  $SiO_4(NH_4)_3H$  remains doubtful. By double decomposition between the alkaline silicates (silicate of soda) and the alkaline earthy salts (Ba, Sr) we obtain precipitates in which the proportion of alkali diminishes with the dilution of the solutions. In the compounds obtained we cannot admit of an isomorphic replacement of the alkaline earths by the alkali. The authors have obtained well-defined compounds by reacting with silica on saturated solutions of baryta water, lime water, or strontium water, at boiling temperature. With a solution of silicic acid containing 1.8 per cent of  $SiO_2$  and baryta water, we obtain a crystalline powder containing  $SiO_3Ba.H_2O$ . Strontium gives the crystalline salt  $SiO_3Sr.H_2O$ , and lime a similar compound of  $SiO_3Ca.H_2O$ , of which the crystalline form has not been proved in a satisfactory manner. By making use of a silicic acid jelly, and the same solutions of  $Ba(OH)_2$ ,  $Sr(OH)_2$ , and  $Ca(OH)_2$  we obtain the same compounds; whatever the excess of alkali used may be, we can only obtain the metasilicates  $SiO_3M_2$ . If we operate in the presence of an excess of acid, we only obtain precipitates of undeterminate composition. By proceeding in the dry way the authors have not been able to obtain sharply defined compounds.—*Zeit. Anorg. Chem.*, vol. xxxiv., p. 455.

## MEETINGS FOR THE WEEK.

- TUESDAY, 24th.**—Royal Institution, 5. "The Solar Corona," by H. F. Newall, F.R.S., &c.
- THURSDAY, 26th.**—Royal Institution, 5. "Literature and the State," by H. G. Wells, B.Sc.
- FRIDAY, 27th.**—Royal Institution, 9. "Progress of Oceanography," by His Serene Highness Albert Prince of Monaco.
- Physical, 5.** "The Law of Action between Magnets and its Bearing on the Determination of the Horizontal Component of the Earth's Magnetic Field with Unifilar Magnetometers," by Dr. C. Chree, F.R.S. "On the ascertained Absence of Effects of Motion through the Ether in Relation to the Constitution of Matter on the Fitzgerald-Lorentz Hypothesis," by Prof. J. Larmor, Sec.R.S. "Coherence and Re-coherence," by Dr. P. E. Shaw and C. A. B. Garrett.
- SATURDAY, 28th.**—Royal Institution, 3. "Spitzbergen in the Seventeenth Century," by Sir William Martin Conway.

CITY OF BIRMINGHAM  
MUNICIPAL TECHNICAL SCHOOL.

The services of an ASSISTANT LECTURER for the CHEMISTRY DEPARTMENT are required from September 1st next.

The commencing salary offered is £125 per annum. The last date for sending in applications is MAY 31st.

Full particulars of the post will be forwarded on application to—  
GEO. MELLOR, Secretary.

Offices of the School, Suffolk Street,  
May 9, 1904.

UNIVERSITY COLLEGE of SOUTH WALES  
and MONMOUTHSHIRE, Cardiff.

The Council of the College invite applications for the following Posts:—

1. ASSISTANT LECTURER in MATHEMATICS.
2. DEMONSTRATOR and ASSISTANT LECTURER in CHEMISTRY.
3. DEMONSTRATOR and ASSISTANT LECTURER in PHYSICS.
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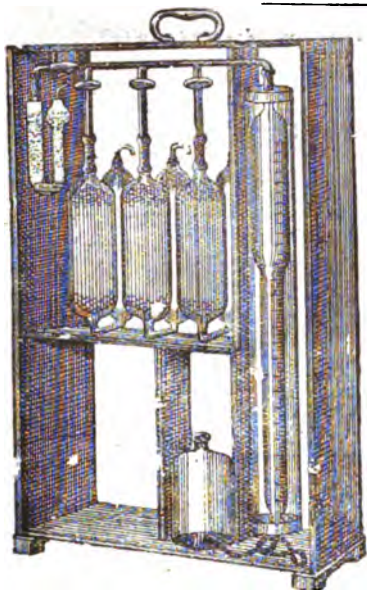
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CONTENTS.

| ARTICLES:—                                                                                                                                          | PAGE |
|-----------------------------------------------------------------------------------------------------------------------------------------------------|------|
| On the Distribution and Spectra of Metallic Vapours in Electric Sparks, by Hugh Ramage, B.A. ....                                                   | 253  |
| Chemical Equilibrium between Ferro- and Ferricyanide of Potassium in the presence of Alkalies, by Maurice Prud'homme ....                           | 254  |
| Determination of the Carbonate of Sodium necessary to Precipitate the Lime and Magnesia for the Chemical Purification of Water, by Leo Vignon ..... | 254  |
| Further Experiments on the Production of Helium from Radium, by Sir William Ramsay, K.C.B., F.R.S., and Frederick Soddy, M.A. ....                  | 255  |
| PROCEEDINGS OF SOCIETIES:—                                                                                                                          |      |
| CHEMICAL SOCIETY .....                                                                                                                              | 258  |
| NOTICES OF BOOKS .....                                                                                                                              | 261  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                         | 261  |
| MISCELLANEOUS .....                                                                                                                                 | 263  |
| NOTES AND QUERIES .....                                                                                                                             | 264  |
| MEETINGS FOR THE WEEK .....                                                                                                                         | 264  |

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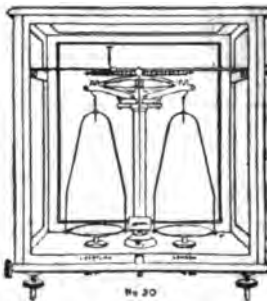
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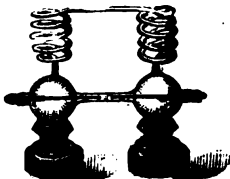
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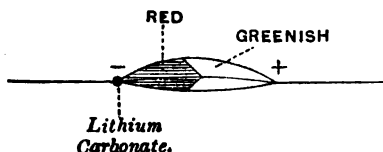
## ON THE DISTRIBUTION AND SPECTRA OF METALLIC VAPOURS IN ELECTRIC SPARKS.

By HUGH RAMAGE, B.A., St. John's College.

Some peculiarities observed in certain lines in the arc and spark spectra of lithium were described by the writer in a paper communicated to the Royal Society in November, 1902 (*Proc. Royal Soc.*, lxxi., p. 164). The blue line, for instance, was found to be broadened in the spectra of the regions near the negative element both in the arc and spark. The broadening of this line was easily observed and photographed in the spectrum of the spark when no Leyden jar was used, and a slight broadening was observed near the positive electrode when no air break was inserted in the secondary circuit and no precautions were taken to keep the electrodes cool. Between these two regions the line was thin and sharp. A further study of electric sparks in air at ordinary pressures has since been made and the results will now be described.

Two clean platinum wires were fixed horizontally in a holder with their adjacent ends about a centimetre apart. When they were connected with an induction coil and the coil was started, the flame surrounding the sparks had the characteristic colour due to the gases of the atmosphere. The anode was then moistened with a solution of a compound of lithium and the coil again started. Flashes of red were observed in the sparks, but when the anode was heated the flame round the sparks became red throughout its whole length, and there were seen in its spectrum the red lithium line and the yellow sodium lines. It would appear from this that some of the lithium and sodium, present as an impurity, were vapourised by the heat and reduced to the free state by the spark, otherwise, according to Gouy's observations (*Comptes Rendus*, lxxxiii., p. 70), no colour should be communicated to a flame in presence of so much free oxygen. A quantity of lithium salt was then fused on the end of one of the wires, forming a globule about 2 m.m. diameter. When the coil was started with this as the anode the red flashes were more frequent and brighter than before, and the orange line of lithium was also observed in the spectrum, but it was extremely feeble. When an air break was inserted in the secondary circuit no red colour was observed as long as the anode remained cold.

On reversing the spark, thus making the globule of lithium compound the kathode, a bright red colour developed in the flame near the kathode, but it did not pass the whole way across to the anode unless the electrodes were brought nearer together.



The appearance of the spark and flame is shown in the figure. There were observed in the spectrum of the red portion, viewed through a vertical slit, the red lithium line as a long line, the orange line as a short line of feeble intensity, and the sodium lines as lines of medium length. The orange line is the strongest line in the subordinate series of lithium, and it is only seen near the central axis of the spark. An exceedingly bright spot of white light

was always observed on the salt where the spark touched it, and in the spectrum of this spot the red lithium line and the yellow sodium lines were somewhat brighter than they were in the red portion of the flame, but the lines of the two subordinate series of lithium were exceedingly bright. It was this spot, and possibly the vapour very close to it, which gave the broadened lines in the diffuse series which were described in the above-mentioned paper. The salt on the kathode becomes hot and sometimes it fuses; if the current is then reversed the flame becomes coloured throughout its whole length, but the colour fades away as the temperature of the bead falls. A single spark passing to the kathode produces both the bright spot and the red flame.

The electrodes were next placed vertically in a line, the bead of salt being first placed on the lower end of the upper electrode. When this was made the kathode, the red colour appeared, but it only extended for about one-half the distance from the electrode that it did when these were horizontal; the upward current of hot air from the spark evidently opposed its motion downwards. The bead and electrodes were allowed to cool and the sparks were then passed in the opposite direction, but no red colour appeared until the bead became heated by the upward current of hot air, and then the red colour appeared and extended down to the clean kathode.

The bead was next put on the upper end of the lower electrode. When it formed the kathode the red colour extended up to the anode, but it was deepest near the kathode. When the electrodes were cold and the bead was made the anode no red colour appeared in the spark.

The lithium vapour liberated from a hot anode would appear to carry a positive charge and thus convey part of the current to the kathode, but the lithium vapour liberated from the kathode does not appear to be charged. In no case has it been observed to be repelled by the anode, and it certainly does not appear to be attracted by it; if it is charged negatively at first the charge must be quickly lost. The lithium would appear to be liberated in definite quantity by each spark, in a very short interval of time, but there is no decisive evidence given by these experiments to show whether the vapour passes away in an explosion wave or merely by diffusion in the gases heated to a high temperature by the passage of the spark. Sodium vapour passes about as far from a kathode of sodium salt as lithium does; this is in favour of the former view.

There is evidently an intense chemical action taking place in the bright spot on the kathode, and there seems no doubt but that lithium is liberated from combination by electrical action at this point. It is probable that the production of the subordinate series is associated with the process of liberation of the metal, that the vibrations producing the lines of these series are peculiar to the atom at the moment of its liberation, perhaps to the atom and the electric charge then given to it, whilst the vibrations producing the lines of the principal series are peculiar to some change, combination or otherwise, of the free atom of the metal. The lines of the subordinate series become broader and apparently brighter as the negative pole of the arc is approached, and this pole, according to Violle, has a temperature of about 2700° when the positive pole is about 3500°, and the temperature of the arc itself is still higher. The apparent brightening of the lines is not a temperature effect.

The results obtained with a compound of sodium are similar to those obtained with lithium. Potassium gives a different result when a bead of its carbonate is on the kathode. There is no flame near the kathode, the spark is reduced to a thin line, whilst it retains its original size and greenish colour near the anode. The bright kathode spot gives the subordinate series as strong lines. Rubidium and caesium resemble potassium, but they give their colours to a portion of the flame near the kathode.

Calcium, strontium, and barium give brilliant colours to the flame near the kathode when beads of their chlorides

are used, and their spectra at the kathode resemble their oxyhydrogen flame spectra, whilst the spectra of the coloured flames are more like those of their Bunsen flame spectra.

Experiments have been made with metallic electrodes of magnesium, aluminium, zinc, tin, and lead. These metals give bright spots on both electrodes and there are differences between the spectra of these. The spectra of the anode spots on magnesium and aluminium contain the "oxide bands" of the metals, but these bands have not been observed in the kathode spots. The writer is continuing the study of this subject in the hope of gaining further knowledge on the constitution of electric sparks and the origin of the spectra.

(NOTE.—Since the above paper was written it has been found by examination of the sparks in a rotating mirror that the coloured flames actually shoot out from the kathode. There was practically no colour given to the spark by the matter thus expelled in an atmosphere of hydrogen; colours were obtained in some instances in nitrogen, but the experiments are not yet concluded. The spectra of the bright spots on the kathodes are always brighter in hydrogen than in air or oxygen, and they are usually brighter under pressures somewhat below the ordinary atmospheric pressure. Zinc and cadmium were reduced from electrodes of their oxides when these were in hydrogen. Their vapours were shot off from the electrode and films of the metals condensed in the form of rings on the tube and on pieces of perforated mica placed in the path of the spark).—*Proceedings of the Cambridge Philosophical Society*, xii., Part 5.

#### CHEMICAL EQUILIBRIUM BETWEEN FERRO- AND FERRICYANIDE OF POTASSIUM IN THE PRESENCE OF ALKALIS.

By MAURICE PRUD'HOMME.

A SOLUTION of ferricyanide of potassium is partially transformed into ferrocyanide when it is submitted to a prolonged boiling with potash. The transformation is integral if we cause the intervention of a reducing body, such as glucose, indigo, &c., which will be oxidised by the oxygen set free.

This property was made use of by Mercer to produce white prints on an indigo blue background. I have examined separately the influence of the quantities of red prussiate, caustic soda, and yellow prussiate on the times necessary for the oxidation or the decolouration of indigo, and have obtained the following results:—

1. The speed of the oxidation of indigo by a mixture of the red prussiate and caustic soda is as nearly as possible proportional to the quantities of the red prussiate and caustic soda used:—

$$V = cR + c', \quad V_1 = c_1S + c'_1.$$

2. The speed of the oxidation of indigo by a mixture of red prussiate, caustic soda, and yellow prussiate is as nearly as possible inversely proportional to the amounts of yellow prussiate when the latter alone is varied.

The fact that the transformation of the red prussiate into the yellow prussiate is not complete, except in the presence of a reducing agent, can be explained by the existence of a state of chemical equilibrium between the elements present:— $\text{Fe}_2\text{Cy}_{12}\text{K}_6 + 2.\text{KOH} \rightleftharpoons 2.\text{FeCy}_6\text{K}_4 + \text{H}_2\text{O}_2$ .

The variations undergone by the speed of the oxidation of indigo, through the presence of the yellow prussiate, corresponds to a phenomenon analogous to that of the etherification of the alcohols, where the addition of an excess of alcohol, acid, ether, or water changes the limit of the chemical equilibrium or of the etherification.

In the equilibrium equation given above, peroxide of

hydrogen is observed; this signifies nothing more than that it is actually formed during the reaction. Nevertheless, it seemed important to examine its action on the yellow and red prussiates, as well as on the indigo itself.

#### Action of Peroxide of Hydrogen on the Prussiates and on Indigo.

1. Neutral peroxide of hydrogen, added in suitable proportion to a solution of yellow prussiate, transforms this latter into red prussiate with the liberation of potash. The solution turns an alcoholic solution of phenolphthalein strongly red, turns red litmus-paper blue, and decolourises indigo blue, but only very slowly, because the amount of caustic alkali present is a minimal quantity.

2. A solution of red prussiate and caustic soda treated with an excess of peroxide of hydrogen changes colour immediately, and is then decolourised, and at the same time it loses all oxidising action on the indigo. The colourless solution, left to itself or heated, gives off oxygen and re-assumes a pale yellow colour; the yellow prussiate is re-formed, and this crystallises if the solution is sufficiently concentrated. There should be produced a colourless combination between the red prussiate and the peroxide of sodium, resulting from the action of the peroxide of hydrogen on the soda. This combination is finally resolved into oxygen and yellow prussiate.

These two inverse reactions (1 and 2) easily explain the state of equilibrium in which an alkaline solution of red prussiate must remain so long as we do not bring in the action of a reducing agent.

3. Peroxide of hydrogen, at about 12 volumes, in the presence of dilute sulphuric acid, slowly decolourised indigo blue. At the temperature of 70° it requires about fifteen hours to decolourise a sample of cotton-cloth containing 0.5 grm. of indigotine to the square metre. Fresh peroxide of hydrogen must be added when the evolution of gas becomes too slow, or even the whole solution may be renewed.

4. In the presence of an alkali, such as soda, the decolouration of indigo by peroxide of hydrogen is much more rapid, and takes place in two and a-half hours at 70°, or five times as quickly as in acid solution. In the cold it takes almost a week to produce decolouration, or sixty times as long as at 70°.—*Bull. Soc. Chim.*, Series 3, vol. xxix., No. 20.

#### DETERMINATION OF THE CARBONATE OF SODIUM NECESSARY TO PRECIPITATE THE LIME AND MAGNESIA FOR THE CHEMICAL PURIFICATION OF WATER.

By LEO VIGNON.

THE method I have described already (*Bull. Soc. Chim.*, Series 3, vol. xxi., p. 559) consists of measuring directly the amount of carbonate of sodium necessary for the chemical purification of water.

In the presence of alcohol and phenolphthalein, the chlorides and sulphates of calcium and magnesium are first transformed into salts of sodium, then the excess of carbonate of sodium is shown by the red colouration of the phenolphthalein; the addition of alcohol accelerates the reaction.

Having applied this method to a very large number of waters, I have accumulated a series of observations which have enabled me to improve it, and to adapt it in the best possible manner to the different cases which occur in practice.

I will sum up the observations made:—

1. The estimation of the carbonate of sodium effected by the method indicated corresponds to the amount of this salt reacting under the conditions of dilution, time, and temperature of the experiment. Let A be this quantity.

2. We can estimate the carbonate of sodium necessary for the complete transformation of the chlorides and sulphates by boiling a known volume of the water under examination in a platinum or nickel crucible with a known amount, in excess, of a titrated solution of carbonate of sodium for thirty minutes. The titration of the mixture after the reaction shows by difference the amount of carbonate of sodium used in the reaction. This amount, B, corresponds with the dilution and temperature (boiling-point), and also with the fact that the carbonate of sodium is in excess. In practice we always find B greater than A; this is explained easily by the fact that in the second case the reaction at boiling-point has been more complete than at the ordinary temperature.

3. By using the colorimetric method, to estimate the excess of carbonate of sodium by phenolphthalein, we observe that after being exposed for some time to light or heat the violet colouration of the liquor disappears gradually. Thus light and heat act on this colouration, and may cause errors if the operation is prolonged.

The action of light and heat is not very strong, however, during the time of the experiment, and the amount of carbonate of sodium found (A) is truly that which corresponds to the reaction of this salt on the chlorides and sulphates of lime and magnesium at the ordinary temperature; but if we leave the substances in contact for twenty-four hours at the ordinary temperature, we find that A increases. Its value approaches that of B by amounts varying for the same time, according to the water examined. By prolonging the contact for several days, and by varying the conditions (ordinary temperature—temperature of 50°; light—darkness), A reaches and surpasses B when the influence of heat or light manifests itself, while A becomes and remains equal to B without surpassing it, after a lapse of time, variable according to the kind of water analysed, when kept at the ordinary temperature and in the dark.

4. If we consider the quantity B, corresponding to the carbonate of sodium, having reacted on the water at boiling-point, in the presence of an excess of this salt, we may ask whether this amount, B, would all enter into the reaction if the exact amount were used without any excess. The last portions of the carbonate of sodium, being very dilute in the mixture, might not enter into the reaction.

Experiment shows that by treating a known water with the amount B, the water remains alkaline, the last portions of the carbonate of sodium not having reacted.

In an experiment the amount B was equal to 280 grms. per cubic metre of water; after boiling for thirty minutes in a nickel crucible, 10 grms. of free carbonate of sodium, remained per cubic metre, or 1/100,000th.

In a second experiment on another water, B being equal to 102 grms. per cubic metre, there remained, after boiling for thirty minutes in the nickel crucible as a result of several trials, from 9 to 20 grms. of free carbonate of sodium per cubic metre of water, or from 9 to 20 millionths.

At each temperature maintained during a given time, we find for each water that a certain quantity, Q, of carbonate of sodium comprised between A (the carbonate of sodium reacting at the ordinary temperature) and B (the carbonate of sodium reacting at boiling temperature in the presence of an excess of carbonate of sodium) should be used in practice. This quantity is that which reacts almost entirely on the chlorides and sulphates, and leaves only a very slight trace of free carbonate of sodium in the water. Practically the free carbonate of sodium should not exceed 10 grms. per cubic metre, or 10 millionths.

The determination of this quantity comprised between A and B should be effected while considering the end aimed at in the chemical purification of the water, and by realising the practical conditions of time and temperature of the operation. If the water is treated at the ordinary temperature, Q will be very close to A; if, on the contrary, the water is for use in boilers, the quantity, Q, will be much closer to B.—*Bull. Soc. Chim.*, Series 3, vol. xxxi. No. 3.

## FURTHER EXPERIMENTS ON THE PRODUCTION OF HELIUM FROM RADIUM.\*

By Sir WILLIAM RAMSAY, K.C.B., F.R.S.,  
and  
FREDERICK SODDY, M.A.

THE research, of which a preliminary account has already been given in the *Proc. Roy. Soc.*, vol. lxxii., pp. 206 and 208, has been continued with the view of ascertaining the volume of emanation produced in a given time from a known weight of radium in the form of bromide, and also the quantity of helium resulting from the spontaneous change of the emanation.

Owing to the minute quantity of material at our disposal, the research has been a somewhat tedious one; but we have succeeded in obtaining fairly concordant measures of the volume of both emanation and helium. The present paper gives a description of the apparatus employed, the methods of experiment, and the quantitative relation between radium and its products.

The inactive nature of the emanation from thorium was the subject of an investigation by Rutherford and Soddy (*Phil. Mag.*, 1902, [6], vol. iv., p. 581). They concluded "that it is a chemically inert gas analogous in nature to the members of the argon family." And they continue: "The speculation naturally arises whether the presence of helium in minerals and its invariable association with uranium and thorium may not be connected with their radio-activity." The discovery was thus the subject of prediction. It followed an attempt to obtain the spectrum of the emanation. Thinking that the spectrum, if brilliant, might be observed by mixing the emanation with a gas of simple spectrum, the first experiments were made by mixing it with helium; but it soon became evident that the helium spectrum overpowered that of the emanation to such an extent as to mask it entirely. And experiments on the removal of gases not belonging to the argon group from the emanation convinced us that its quantity was so small as to require special contrivances in order to deal with it. All apparatus, consequently, was constructed on a minute scale of capillary tubing, less than half a millimetre in diameter. Approximate measurements of the scale of the apparatus in centimetres are given in the sketches.

Fig. 1 gives a sketch of the first apparatus, which was soon abandoned; suffice it to say that an attempt was made to accumulate the emanation in A, which contained a solution of several grms. of impure chloride, obtained from very impure carbonate, and to examine its spectrum in the U-tube B, made of capillary tubing, with electrodes of platinum as shown. The spectrum was that of carbon monoxide and dioxide.

*Experiment 1.*—A brief description of this experiment has already been given (*Proc. Roy. Soc.*, lxxii., 206). Twenty milligrams of radium bromide was dissolved by admitting water boiled *in vacuo* to the crystals in the bulb A (Fig. 1), which had previously been freed from air with the pump. The bromide, as a letter from the seller informed us, had been prepared in the solid state about two and a-half months. The evolved electrolytic gas containing the emanation was collected through the pump and introduced into the apparatus, of which a sketch is given in Fig. 2. Before this was done, the whole apparatus had been exhausted, and washed out with oxygen several times, admitted through the gas-burette. The emanation, too, was collected in a tube which had been used for oxygen, the object being to keep nitrogen out of the tubes, and so to avoid its spectrum, which is difficult to remove.

The gas, of which there was about half a c.c., was admitted into the gas-burette through the inverted syphon A; the stopcock being reversed, it was passed slowly into the tube B, which contained a spiral of thin, partially oxidised copper wire, and which had previously been exhausted; during the introduction of the gas the copper

\* A Paper read before the Royal Society, April 28, 1904.

spiral was kept red-hot by a current. The water produced was absorbed in the tube c, which contained phosphorus pentoxide. Mercury was then admitted into b and c, so as to displace the gas through the stopcock d, which was then shut. The vacuum tube f had been previously glowed out until phosphorescent. This vacuum tube is represented in natural size in Fig. 3; its capacity was about one-third of that of the U-tube and accessory tubing. The spectrum of carbon dioxide was alone seen. With a jar and spark-gap interposed, on comparing the spectrum with the jar discharge in a similar tube containing carbon dioxide, a yellow line was visible in the gas from radium, and also a bright blue line, absent in the spectrum of the pure dioxide. The spectrum of helium was then thrown in through a comparison prism, when no doubt remained that the yellow line was actually  $D_3$ . By cooling the U-tube, the emanation and dioxide were condensed, and the helium spectrum increased greatly in brilliancy. After half-an-hour the tube was sealed off. The position of the  $D_3$  line was con-

approximate wave-length 6145 and 5675, the former faint but distinct, the latter moderately bright. The vacuum-tube did not glow visibly in the dark, showing that the emanation had been almost completely removed. The U-tube was next placed in communication with the pump, still surrounded with liquid air, but no gas could be extracted; now the U-tube had probably two or three times the capacity of the vacuum-tube; and at the low temperature of liquid air, almost twenty times the quantity of helium must have remained in it. It shone brilliantly in the dark. The passage to the pump was then closed, and the liquid air removed. On again establishing communication, a brilliant phenomenon was observed in the dark; the glowing emanation passed through the capillary somewhat slowly, rushed along the wider connecting tubing, was delayed in passing through the tightly packed phosphorus pentoxide, and finally filled the barrel of the Töpler pump. On raising the reservoir, the gas grew more luminous as its volume was decreased, and on

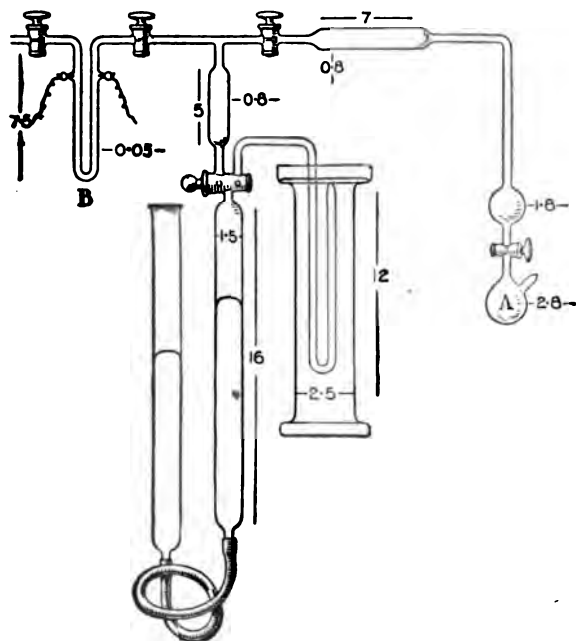


FIG. 1.

firmed to within one-tenth of the distance between the two sodium lines,  $D_1$  and  $D_2$ .

**Experiment 2.**—A second apparatus similar to the last was made of entirely fresh glass, so as to exclude any possibility of contamination with helium; and the observation was repeated with 31.8 milligrams of radium bromide, kindly lent by Prof. Rutherford, which had been kept in the solid state at least three months. The apparatus was slightly modified, as shown in the dotted lines in Fig. 2, so as to avoid taking the gas through the pump. As before, the whole apparatus was washed out with oxygen, and the copper spiral was glowed in oxygen, so as to oxidise it superficially, and so render it able to deal with the excess of hydrogen, as well as with the constituents of the water which had been decomposed. After the gas had been admitted by opening the stopcocks g and h, the copper spiral was kept glowing for three-quarters of an hour. The U-tube was then cooled with liquid air, and tap d was opened.  $D_3$  was seen. Mercury was admitted to the tubes b and c, and the vacuum-tube was sealed off. It now showed all the visible helium spectrum except the faint least refrangible red, as well as the yellows, green, and violet of mercury. Two unidentified lines were also measured, of

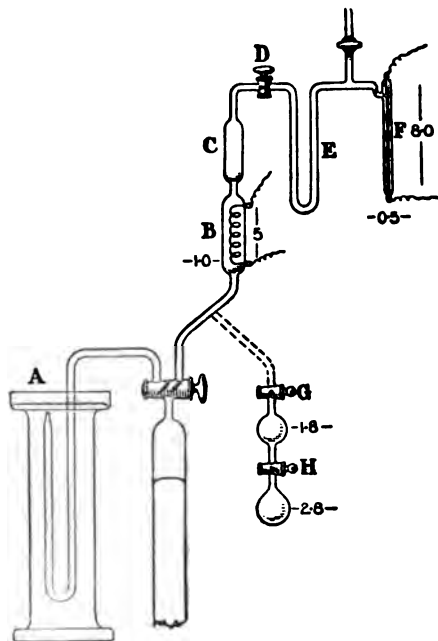


FIG. 2.

lowering it, the glowing gas appeared to lie on the surface of the mercury for a fraction of a second, falling with the falling mercury; but it soon spread through the whole barrel by diffusion.

The bubble of gas pumped out was treated with a drop of caustic potash, when a considerable fraction was absorbed. By next day the volume of the bubble had increased.

Inasmuch as all samples of emanation showed the spectrum of carbon dioxide, the presence of which was believed to be due to the oxidation of the tap-grease, an apparatus was constructed in which the use of taps was, as much as possible, avoided. All the emanation from about 60 milligrams of radium bromide was introduced into the burette A, the only gas present being oxygen. From the burette it passed through the bulb B, which contained concentrated potash solution; it then passed through C, which was charged with solid potash and was deprived of moisture by contact with phosphorus pentoxide in D. The level of the mercury in the trap was at E, so that the emanation reached the spiral G, cooled with liquid air; the whole of the emanation was washed into the spiral by admission of a little pure oxygen from A; on exhaustion with

the pump the gas was not luminous, showing that the emanation had been almost completely retained in the spiral. Mercury was then allowed to rise in the trap until the bulb F was filled; the connection to the pump was then sealed at H and the spiral allowed to warm up. The emanation in the vacuum tube showed a bright green spectrum, but on filling the spiral with mercury and sealing off the vacuum tube, the spectrum of carbon dioxide became visible; D<sub>3</sub> was not seen.

Next day this line was seen, but very feeble; its strength increased from day to day, and in five days the yellow, green, and two blues were visible as well as the violet; their identity was proved by means of a comparison spectrum.

Subsequent experiments were made in which the heated spiral of copper was replaced by a tube containing a fragment of phosphorus; the emanation was washed out of

19.48 c.c.; oxygen, 10.37 c.c.; nitrogen, 1.02 c.c. = 30.87 c.c.

The nitrogen was manifestly derived from leakage; after deducting one-fourth of its volume of oxygen, the remaining gas has practically the composition of electrolytic gas. The rate of accumulation is about  $\frac{1}{2}$  c.c. a day.

The object of the experiment, of which an account will now be given, was to form an estimate of the amount of helium produced by comparing the intensity of its spectrum with that of a known quantity of helium at a known pressure.

*Experiment 3.*—This gas was exploded and left a residue of nitrogen; it was then mixed with a large excess of oxygen, and sparked in presence of caustic soda for some hours to remove nitrogen. The oxygen was next withdrawn by means of phosphorus, and the minute bubble

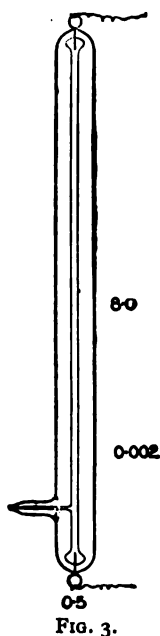


FIG. 3.

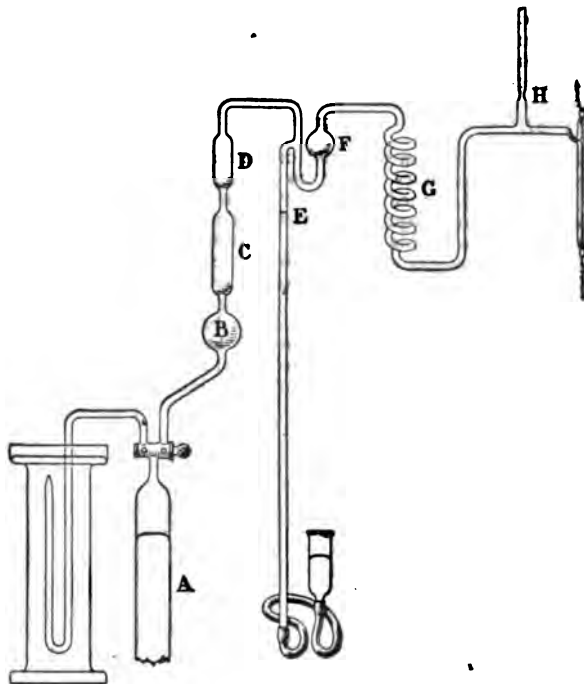


FIG. 4.

the condensing tube by a few bubbles of oxygen. The bulb of potash solution was retained, but the solid potash was replaced by solid barium hydroxide. This plan was not so effective in removing carbon dioxide, yet on keeping the tube for three days, and condensing the carbon dioxide with liquid air, D<sub>3</sub> was easily visible, although weakened by the spectrum of carbon monoxide.

On two subsequent occasions the gases evolved from both solutions of radium bromide were mixed after four days' accumulation, which amounted to about 2.5 c.c. in each case, and were examined in a similar way. In this case the non-condensable gases alone were examined, the emanation being retained. Whereas with the emanation almost the whole can be introduced into the vacuum tube, with permanent gases only about one-twentieth part is available for the purpose of the spectrum. The D<sub>3</sub> line of helium could not be detected.

The vacation now intervened, and the bulbs containing the dissolved radium bromide were connected with a mercury reservoir and with a gauge, so that the pressure should not rise and burst the bulbs. The gas accumulated during sixty days; its composition was: Hydrogen,

left was mixed with a bubble of oxygen, in order to wash it into the apparatus to which the vacuum tube was sealed. As already described, this apparatus did not differ from that shown in Fig. 2, except for the fact that the tube containing the copper spiral was replaced by one containing a fragment of phosphorus, in order to withdraw the oxygen. The phosphorus was warmed and withdrew the oxygen. The gas was then forced by means of mercury through a cooled U-tube, and a portion reached the vacuum tube. On passing a current for an instant, the D<sub>3</sub> line was plainly seen, but nitrogen was also present in small amount. The tube was then sealed off.

The volume of the spectrum tube, including the U-tube, had been previously estimated by filling it twenty times with air and pumping out each time; from this measurement the total volume was found to be 0.310 c.c., and after the U-tube had been sealed off, the same process was repeated with the U-tube and connections, minus the spectrum tube. The volume of the spectrum tube was thus found to be 0.165 c.c.

An exactly similar spectrum tube made of the same glass and having the same length was attached to a bulb



tube, from which it could be cut off by turning a stopcock; the bulb tube in its turn could be cut off from the pump by a stopcock. The capacity of the spectrum tube as well as of the bulb tube was known. A known amount of helium was introduced into the bulb tube and the spectrum tube by means of an inverted syphon furnished with two stopcocks; the volume between the stopcocks was 0.0268 c.c. As the volume of the spectrum tube and connections was 1.68 c.c., and that of the bulb was 1.25 c.c., when the gas contained in the spectrum tube was allowed to expand into the evacuated bulb tube, its volume was reduced in the ratio  $1.68/1.25 + 1.68$ , or 0.57. The spectrum tube containing the fraction of the helium from 60 days' accumulation was placed in series with that containing helium, so that the same current traversed both, and their spectra were compared as regards luminosity of the  $D_3$  line. It was necessary to divide the contents of the helium tube seven times before the  $D_3$  line could be regarded as of comparable intensity in both spectrum tubes. Multiplying this ratio by the volume of the helium admitted at atmospheric pressure into the apparatus, the volume remaining in the apparatus after rarefaction is given:—

$$(0.57)^7 \times 0.0268 = 0.000517 \text{ c.c.}$$

Now the volume of the helium tube and connections was by chance practically ten times that of the spectrum tube alone (1.65 and 0.165), hence the spectrum tube contained 0.000052 c.c., or 0.052 cub. m.m. The total quantity obtained was twice this amount, or 0.1 cub. m.m.

As 1 litre of helium weighs 0.18 gm., for its density is twice that of hydrogen, 0.1 cub. m.m. weighs 0.000018 m.grm. This amount is the product of 50 m.grms. of radium bromide in 60 days; hence, 1 gm. of the bromide should give in a year 0.0022 m.grm. It should be mentioned that the spectrum of argon was present, and it may have seriously interfered with this estimation. The helium, too, may have penetrated and been retained in the glass.

**Experiment 4.**—It appeared feasible to attempt to measure the actual volume of the emanation in a fine capillary tube. Thinking that any bought capillary tube would be too wide, we drew a very narrow one, which had an electrode sealed into its end. It turned out, however, to be very irregular, and the results as regards volume are not very trustworthy. A is the capillary tube, with a platinum electrode of very fine wire sealed into its upper end; the mixed hydrogen and oxygen containing the emanation were introduced into the explosion burette F through the inverted syphon E; some moist caustic potash had been melted in the top of the burette, so as to remove from the gases any possible carbon dioxide which might have been produced by the flame causing any organic dust in the burette to burn. After the gases had been exploded, the excess of hydrogen, together with the emanation, was allowed to stand for some time in contact with the caustic potash. The upper part of the apparatus having been completely evacuated, the connection with the pump was closed, and the tube leading to the reservoir of the burette was clipped; on making communication by turning the tap of the burette, the hydrogen and the emanation entered the apparatus. Liquid air was then poured into the tube C, so as to cool the bulb B, where the emanation condensed. After raising and lowering reservoir of the burette several times, in order to convey the emanation into the bulb B, the tap of the burette was closed, and that leading to the pump opened. Again opening cautiously the tap of the burette, the mercury was allowed to rise, passing through the tube D containing phosphorus pentoxide as far as G; the evacuation was then completed until not a trace of a bubble passed through the pump. The tap leading to the pump was closed, and that of the burette opened, until the mercury had risen to near the bulb B. On darkening the room the bulb B was brilliantly luminous; indeed, it was possible to read a watch by its light. The liquid air was allowed to evaporate away, and the reservoir of the burette

lowered and its tap opened; by gently raising the reservoir the emanation was all collected in the capillary tube A. The volume of the emanation was read from day to day by help of a reading telescope. It contracted regularly; the tube was coloured deep purple after some days, and this made reading difficult, but by a brilliant illumination behind the rise of the mercury could be followed. No attempt was made to pass a discharge for twenty-eight days; after that lapse of time the emanation had contracted to a volume occupying only 0.1 of a m.m. of the capillary tube at a pressure of about 50 m.m., yet it maintained its brilliancy till the very last; only the length of tube illuminated grew shorter and shorter. On freezing out the mercury vapour by cooling the bulb B with liquid air, the

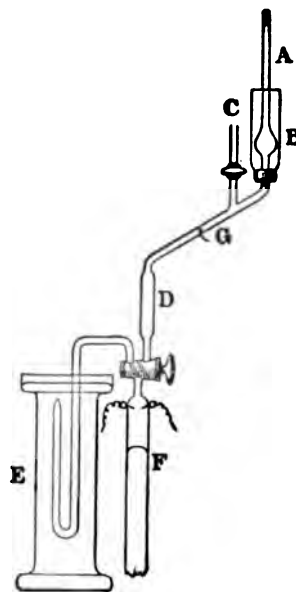


FIG. 5.

helium spectrum was visible, and at the same time the effect of passing the discharge was to reproduce gas in the capillary tube.

After the conclusion of the experiment, the tip of the tube was cut off immediately below the platinum wire, and the capillary depression was measured at different levels. The capillary tube was then cut off, and the volume determined by weighing with mercury; it was then calibrated by a shifting thread of mercury, under a reading microscope. The final results were:—

| Time.               | Volume.         |
|---------------------|-----------------|
|                     | 0.124 cub. m.m. |
| Start .. .. .       | 0.027 "         |
| One day .. .. .     | 0.011 "         |
| Three days .. .. .  | 0.0095 "        |
| Four days .. .. .   | 0.0063 "        |
| Six days .. .. .    | 0.0050 "        |
| Seven days .. .. .  | 0.0041 "        |
| Nine days .. .. .   | 0.0020 "        |
| Eleven days .. .. . | 0.0011 "        |
| Twelve days .. .. . | 0.0004 "        |
| Four weeks .. .. .  |                 |

The comparatively large volume at the start is very remarkable; we can only record it, it may possibly have been due to the mercury sticking in the capillary tube, which was narrower below.

(To be continued).

# PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, May 5th, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

(Concluded from p. 247).

79. "The Resin Acids of the Coniferae. Part I. The Constitution of Abietic Acid." By THOMAS HILL EASTERFIELD and GEORGE BAGLEY.

The resin acids of the Coniferae as a class have the following points in common:—(1) They readily become superfused, and then set to a resin or glass, which crystallises if fused and maintained for some time at a temperature slightly above the initial melting-point; (2) they nitrate without difficulty in glacial acetic acid solution; (3) in a fused state, they readily absorb oxygen from the air; (4) their esterification velocity is extremely low; (5) with hydriodic acid, they yield hydrocarbons of nearly the same composition as the diterpenes, with which they have erroneously been regarded as identical.

Colophony, on distillation under diminished pressure or in superheated steam, yields a product which gives analytical results agreeing accurately with those required for abietic acid (m. p. 160—165°).

By the slow distillation of abietic acid, even under reduced pressure, a hydrocarbon,  $C_{18}H_{28}$ , is produced, which is undoubtedly the "colophene" obtained by Deville on distilling colophony, but is not the "colophene" which the same author produced by polymerising turpentine by means of sulphuric acid. In order to avoid ambiguity, the name "abietene" is proposed for the compound derived from abietic acid.

Krämer and Spilker, by distilling colophony under pressure, obtained a hydrocarbon to which they assigned the formula  $C_{18}H_{28}$ , regarding it as being derived from abietic acid by the loss of carbon dioxide.

Abietene,  $C_{18}H_{28}$ , boils at 199—200° (13 m.m.), 247—250° (82 m.m.), 340—345° (760 m.m.), has a sp. gr. 0.973 at 19°/19°, and  $n_D^{20}$  1.537 at 20°; it is thus identical with the "diterbenthyl" ( $C_{20}H_{30}$ ?) which Renard (*Comptes Rendus*, 1887, cv., 865) isolated from resin oil.

Abietene is produced when abietic acid is heated with fuming hydriodic acid at 200°; the gases formed at the same time have been analysed, and were found to consist of 90 per cent of carbon monoxide and dioxide approximately in the ratio  $CO : CO_2$ , the volume of oxides of carbon formed after two hours' heating being 80 per cent of that required for the elimination of one carboxyl group from abietic acid.

Abietene, when distilled with one-third of its weight of sulphur, yields a small quantity of retene (m. p. 99°), but with twice this proportion of sulphur an isomeric hydrocarbon is obtained melting at 86°; at the same time, a hydrocarbon boiling at 330—360° (30 m.m.) is produced, which is still under examination. If the distillation with sulphur is conducted under reduced pressure, retene is the principal product. These two hydrocarbons are also formed by distilling Merck's "retene puriss." with one fifth of its weight of sulphur.

The foregoing observations support the view that hydrogenated retenes are probably normal constituents of resin oil (*Ber.*, 1899, xxii., 3368; 1903, xxxvi., 647), and that resin oil is undoubtedly a hydrogenated retene.

This conclusion is further strengthened by the observation that abietene, when reduced with hydriodic acid and excess of phosphorus at 240°, takes up two atoms of hydrogen and yields a hydrocarbon, dihydroabietene, which appears to be identical with the dodecahydroretene obtained by Liebermann and Spiegel (*Ber.*, 1889, xxii., 779) by reducing retene under similar circumstances.

The above observations lead to the conclusion that abietic acid is decahydroretene-carboxylic acid, but the

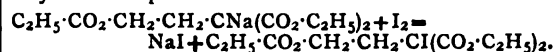
accepted formula for retene would not account for its low esterification velocity. There is, however, no experimental evidence for the assumed para-position of the methyl and isopropyl groups in retene. That these groups are really in the meta-position in abietic acid and in retene is rendered probable by the observation of Kolbe (*Ber.*, 1880, xiii., 888) that resin spirit is rich in *m*-cymene, but contains only small quantities of ordinary cymene.

80. "The Additive Products of Benzylideneaniline with Ethyl Acetoacetate and Ethyl Methylacetoacetate." By FRANCIS ERNEST FRANCIS and Miss MILLICENT TAYLOR.

It was shown that the additive product of benzylideneaniline and ethyl methylacetoacetate exists in one form only, being unaffected in solution by traces of either piperidine or sodium ethoxide. This confirms Schiff's view of the constitution of such substances. The various data in connection with the additive products from ethyl acetoacetate and benzylideneaniline were indicated, and Morrell and Bellars' work on the subject was criticised.

81. "Studies on Ethyl Carboxyglutarate. Part I. Action of Halogens on Ethyl Sodiocarboxyglutarate." By OSTWALD SILBERRAD and THOMAS HILL EASTERFIELD.

When halogens were allowed to act on ethyl sodiocarboxyglutarate, no condensation occurred, but instead of ethyl carboxyglutarate the haloid derivatives of the original ethyl carboxyglutarate were formed. The reaction may be thus represented—



The products proved to be identical with the compounds subsequently obtained by the direct halogenation of ethyl carboxyglutarate.

82. "Studies on Optically Active Carbimides." Part I. By ALLEN NEVILLE and ROBERT HOWSON PICKARD.

The authors have investigated (i.) the preparation of some optically active carbimides, (ii.) the use of such compounds for the resolution of synthetical alcohols and amines containing asymmetric carbon atoms, (iii.) the measurement of the velocity of reaction between such compounds and various alcohols and amines with the object of contrasting the influence of (1) the constitution of the alcohols and amines, (2) the temperature, (3) the solvents, and (4) various catalytic agents on the velocity of such reactions.

The compounds described were:—Bornylcarbimide (compare Forster and Attwell, *Proc.*, vol. xx., p. 91), ethyl bornylcarbimate and dibornylcarbimide, 1-menthylcarbimide, methyl ethyl, and propyl 1-menthylcarbimates, and di-1-menthylcarbimide.

83. "The Comparison of the Rotation Values of Methyl, Ethyl, and *n*-Propyl Tartrates at Different Temperatures." By THOMAS STEWART PATTERSON.

Methyl tartrate was first shown to be capable of existence in a solid form melting at 61.5°.

Data for the variation of rotation with change of temperature of methyl, ethyl, and propyl tartrates were given. Comparisons of the rotation values of these substances at identical temperatures were shown to be of little value, especially at low temperatures. Since, however, the rotation of methyl tartrate vanishes at 0°, that of ethyl tartrate probably at -34°, and that of propyl tartrate probably at -60°, it may be assumed that the three substances are in corresponding optical conditions at these temperatures, and, in general, methyl tartrate at T°, ethyl tartrate at (T - 34°), and propyl tartrate at (T - 60°) will also be in corresponding conditions as regard rotation. It is shown that comparisons of rotation effected at corresponding temperatures are much more satisfactory than those obtained for identical temperatures, and that if the rotations are taken at corresponding temperatures, the increment of  $2CH_2$  in passing from methyl to ethyl tartrate rather more than doubles the rotation, whilst the next increment of  $2CH_2$  in passing to propyl tartrate increases the rotation to 1.41 times that of ethyl tartrate. The

rotation of propyl tartrate is therefore almost three times that of methyl tartrate. These results hold within wide limits of temperature.

84. "Note on the Action of Hydrogen Sulphide on Formaldehyde and Acetaldehyde Solutions." By JULIEN DRUGMAN and WILLIAM ERNEST STOCKINGS.

Although the conditions under which various thio-derivatives are formed when hydrogen sulphide reacts with aqueous solutions of formaldehyde and acetaldehyde respectively have been thoroughly investigated by Hofmann (*Ber.*, 1868, i., 176; 1869, ii., 152; 1870, iii., 584), Pirner (*Ber.*, 1871, iv., 257), Klinger (*Ber.*, 1876, ix., 1894; 1877, x., 1879; 1878, xi., 1023), Baumann and Fromm (*Ber.*, 1889, xxii., 2600; 1890, xxiii., 69; 1891, xxiv., 1434, 1457), and others, it is not generally known that, provided hydrochloric acid or other strong mineral acid is absent, the reaction affords a sure means of detecting formaldehyde, even in the presence of acetaldehyde or other higher fatty aldehydes.

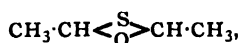
1. When a dilute aqueous solution of formaldehyde is saturated with hydrogen sulphide in the absence of hydrochloric acid, and the test-tube set aside in a warm place (30–50°), no visible change occurs for about three or four hours, but afterwards a white, amorphous, flocculent precipitate is gradually deposited. This substance, when dried in a desiccator over sulphuric acid, melts, with decomposition, between 85° and 110°.

This characteristic thio-derivative is precipitated from formaldehyde solution by hydrogen sulphide even in the presence of acetaldehyde, methyl, and ethyl alcohols, acetone, or acetic acid; the melting-point of the product formed in presence of acetone or acetic acid is fairly definite, namely, 98–103°, and sometimes quite sharp at 98°. According to Baumann (*loc. cit.*), the compound has the composition  $3(\text{CH}_2\text{S})\cdot\text{CH}_2\text{O}$ ; it is distinguished from the odourless, crystalline trithioformaldehyde,  $(\text{CH}_2\text{S})_3$ , both by its lower melting-point and strong odour. The presence of even 0.1 per cent of formaldehyde in aqueous solution may be easily detected by means of this reaction.

2. The crystalline trithioformaldehyde,  $(\text{CH}_2\text{S})_3$ , first prepared by Girard by the reduction of carbon disulphide by means of nascent hydrogen (*Comptes Rendus*, 1870, lxx., 623), and afterwards fully investigated by Hofmann (*loc. cit.*), results when hydrogen sulphide acts on a solution of formaldehyde previously strongly acidified with hydrochloric acid. The crude product melts indefinitely between 180° and 205°, but it crystallises from hot acetone in white needles which melt at 216°.

3. When a solution of acetaldehyde is saturated with hydrogen sulphide in the absence of hydrochloric acid or other strong mineral acid, and the test-tube set aside in a warm place, the liquid soon assumes a milky appearance, owing to the separation of minute, oily particles, which gradually collect in drops on the bottom and sides of the test-tube, leaving the rest of the liquid perfectly clear and translucent. No solid product is ever obtained under these conditions, a circumstance which at once differentiates the behaviour of acetaldehyde from that of formaldehyde.

4. Several distinct crystalline thio-derivatives can be obtained by the action of hydrogen sulphide on aqueous or alcoholic solutions of acetaldehyde previously acidified with hydrochloric acid. Among these may be mentioned the volatile bimolecular compound,—



which melts at 61°, and the  $\alpha$ -,  $\beta$ -, and  $\gamma$ -trithioacetaldehydes,  $(\text{C}_2\text{H}_4\text{S})_3$ , which melt at 101°, 125–126°, and 76° respectively. The character of the products and the relative proportions in which they are formed depend entirely on the experimental conditions. When, however, a dilute (1 to 20 per cent) aqueous solution of acetaldehyde, acidified with about one-fifth of its volume of strong hydrochloric acid, is saturated with hydrogen sulphide, and the test-tube set aside in a beaker over a water-bath kept at

50–60°, a very characteristic and striking phenomenon is observed. At the outset, the liquid assumes the milky appearance described under (3), then gradually becomes clear, and finally, after some hours, a volatile thio-compound sublimes on to the cooler portions of the tube in the form of long, glistening needles, which, after crystallisation from acetone or dilute alcohol, melt at 75–78°. The sublimate is not, however, formed if the original solution contains formaldehyde as well as acetaldehyde. In this case, the mixed thio-compounds separate together from the liquid as a crystalline precipitate. Moreover, if the original solution contains a volatile solvent, such as acetone or alcohol, the sublimate will only appear after the solvent in question has nearly all evaporated.

5. Aqueous solutions of propaldehyde or isobutaldehyde saturated with hydrogen sulphide, without any addition of hydrochloric acid and set aside in a warm place, exhibit a similar behaviour to that of acetaldehyde solutions under the same conditions. The oily thio-compounds obtained soon fall to the bottom of the test-tube, leaving the supernatant liquid quite clear. The presence of hydrochloric acid does not materially alter the character of the appearances observed, except in the case of isobutaldehyde, when, after a very long time, a slight crystalline sublimate may be formed.

85. "The Viscosity of Liquid Mixtures." By ALBERT ERNEST DUNSTAN.

The following conclusions were drawn from the author's investigation on the viscosity of liquid mixtures:—

1. Aqueous solutions give abnormal results. This is true for all known cases, which, however, are always solutions of two associated liquids, as, for example, alcohol and water.

2. Whenever chemical affinity is existent to any marked extent in the two liquids of a mixture, abnormal results occur. In many such cases, definite complexes have been isolated.

3. Whenever marked abnormalities present themselves, it is noted that they do so near points of definite molecular composition, but these points of abnormality are not constant, and shift with change of temperature. At a sufficiently high temperature, they would probably vanish, as then no formation of complexes would be possible.

4. When points of minima are obtained in the viscosity curve, it is difficult to say whether association or dissociation is proceeding. It certainly seems due to some change in molecular aggregation.

5. Substances containing the hydroxyl group (and associated) have a higher viscosity, as a rule, than monomolecular substances. Hence the rise of viscosity in the case of alcohol—water is probably due to the formation of complexes.

A further examination of hydroxylic substances is in progress.

86. "The Conversion of Isopropyl Alcohol into Isopropyl Ether by Sulphuric Acid." By FRANK SOUTHERDEN.

In the course of some experiments on etherification by the agency of small quantities of sulphuric acid at high temperatures, it has been found possible to prepare isopropyl ether. The yield is very poor, and therefore without improved conditions the method cannot be said to compete with that involving the use of alkyl iodide (Erlenmeyer, *Annalen*, 1863, cxxvii., 305), but it is interesting as an example of the production of a secondary ether from its alcohol.

Previous observers have obtained only propylene in this reaction. When, however, 3 c.c. of  $\text{H}_2\text{SO}_4$  were employed for 100 c.c. of isopropyl alcohol (b. p. 81–83°), and the mixture heated for six to twelve hours at 150–160°, the ether was formed along with propylene. After removal of the unchanged alcohol by prolonged treatment with sodium, the whole distilled at 70–70.5° (uncorr.). Zander (*Annalen*, 1882, ccxiv., 164) gives 68.5–69° as the boiling-point of the ether. The isopropyl ether has an intense odour resembling peppermint; bromine in carbon tetrachloride

solution is not decolorised by it. Two vapour density determinations obtained by Victor Meyer's method gave 51.3 and 51.4, whereas the calculated value for  $(C_3H_7)_2O$  is 51.

## NOTICES OF BOOKS.

*A Safe Course in Experimental Chemistry.* By W. T. BOONE, B.A., B.Sc. The University Tutorial Series. London: W. B. Clive. 1904. Pp. 180.

WE are not sure that the author has been very happy in his choice of a title for this book; it appears from the preface that the experiments "could with few exceptions be performed at home with perfect safety"—and hence we presume the title, "A Safe Course"; but is not this accentuating rather unnecessarily the very slight amount of danger attached to a simple course of experimental chemistry for beginners?

As for the arrangement and compilation of the book, we have no criticism to offer. It is well adapted for its avowed purpose, as set forth in the syllabus issued by the Incorporated Association of Head-masters, viz., "to train students to solve simple problems by experiment, to work accurately, and with a clearly defined purpose, and to reason from observation." The book has a good index.

*Experiments on the Metabolism of Matter and Energy in the Human Body, 1900-1902.* By W. O. ATWATER, Ph.D., and F. G. BENEDICT, Ph.D. Washington: Government Printing Office. 1903. Pp. 357.

THE experiments here reported form part of a series which is in progress at Middletown, Conn., in co-operation with the Storrs Agricultural Experiment Station and Wesleyan University. The present Bulletin includes, along with the details of the experiments, an account of a number of important improvements in apparatus and method not hitherto described.

The experiments yield valuable data regarding the conservation and transformation of matter and energy in the body, the demands of the body for nutriment, the effects of muscular work upon such demand, and the actual nutritive values of different food materials and their ingredients.

The results of these experiments are summarised with those of thirty-four previously reported, making fifty-five experiments in all.

The whole inquiry rests upon the principle that the transformations of matter and energy in the body take place in accordance with the two fundamental laws of the conservation of mass and the conservation of energy; and the conclusion drawn from the experiments is that the law of the conservation of energy held good as regards the men who were studied. For practical purposes, therefore, we are warranted in assuming that the law obtains in general in living organisms, as there is every reason to believe that it must.

*Wilhelm Ostwald.* By P. WALDEN. Leipzig: Wilhelm Engelmann. 1904.

LAST year saw the publication of more than one volume in celebration of Prof. Ostwald's jubilee, of which books this short biography is a very good specimen. It contains a graphic picture of the life and character of a man of the most marked individuality. The difficulty of the task with which the biographer has to grapple is very materially augmented owing to the fact that the great master is happily still with us, but the author has satisfactorily coped with this difficulty, and has produced a very attractive and appreciative little volume. The short section on Ostwald's

artistic talents will probably contain much that is new to many to whom he is not known personally, a heliogravure of one of his pastels as well as an excellent portrait being included.

*Grundlinien der Anorganischen Chemie.* ("The Principles of Inorganic Chemistry"). By W. OSTWALD. Second Edition. Leipzig: Wilhelm Engelmann. 1904.

THE facts that in three years the first edition, consisting of 4000 copies, of this work has been exhausted, and that English and Russian translations have appeared, show plainly that Ostwald's views regarding instruction in pure chemistry have strongly recommended themselves to a large number of teachers and lecturers in spite of a good deal of vehement opposition at one time.

In this edition no fundamental alterations have been made. Beyond carefully revising the whole of the text and re-modelling in places where greater detail of explanation seemed necessary, the author has been content to issue the work in the same form as at first, and there can be no doubt that the more this method of introducing the learner to the study of chemistry is known the greater will be the demand for this text-book.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 17, April 25, 1904.

**Atomic Weights of Oxygen and Hydrogen and the Probable Value of their Atomic Relation.**—Ph. A. Guye and Ed. Mallet.—The final value resulting from the authors' careful determinations is  $O = 15.8787$  given  $H = 1$ , or  $H = 1.00764$  for  $O = 16$  (with an error of  $1/80,000$  only on the most discordant of the mean values). The weight of a litre of normal oxygen ( $1.42886$  grm.) and of hydrogen ( $0.089875$  grm.) is also determined.

**Experimental Researches relative to certain Cyclic Amines.**—P. Lemoult.—The author measures experimentally the heats of combustion of the following cyclic amines:—

|                             |                        |              |
|-----------------------------|------------------------|--------------|
| Xylidine . . . .            | At constant volume . . | 1111.42 cal. |
|                             | " pressure . .         | 1112.70 "    |
| Monoethylaniline . .        | " volume . .           | 1125.60 "    |
|                             | " pressure . .         | 1126.88 "    |
| Anisidine . . . .           | " volume . .           | 927.29 "     |
|                             | " pressure . .         | 928 "        |
| $\alpha$ -Naphthylamine . . | " volume . .           | 1268.78 "    |
|                             | " pressure . .         | 1269.78 "    |
| $\beta$ -Naphthylamine . .  | " volume . .           | 1266.5 "     |
|                             | " pressure . .         | 1267.5 "     |

**Formation of Hydrogen Silicide,  $SiH_4$ , by Direct Synthesis from its Elements.**—A. Dufour.—Hydrogen and silicon unite directly, but in very small quantities, at a temperature above that of fusion of silicon, giving hydrogen silicide,  $SiH_4$ . This result is in agreement with the endothermic formation of hydrogen silicide.

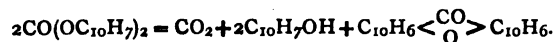
**Lead-aluminium Alloys.**—H. Pécheux.—The author prepares three alloys of lead and aluminium, containing 93, 95, and 98 per cent of aluminium respectively. He finds the density of these and examines their properties. They are all unattacked by moist air at their melting-points, but are readily attacked by most acids. Distilled water has no action on them either at  $0^\circ$  or  $100^\circ$ .

**Colloidal Gold.**—M. Hanriot.—Henrich described, under the name of colloidal gold, solutions which he obtained by treating gold chloride by certain reducing phenols, such as pyrocatechin and hydroquinone. The author prepares one of these substances from pyrocatechin in the form of a blue powder, slightly soluble in pure water, but insoluble in a slight excess of sulphuric or nitric acid. It dissolves in alkalis easily, especially in ammonia. An analysis of the substance dried at 40° shows—Water driven off at 100°, 2.04; water lost at a red heat, 6.31; gold (direct estimation), 91.53; SO<sub>3</sub>, 0.39. He discovers also the curious fact that colloidal gold does not dissolve in mercury.

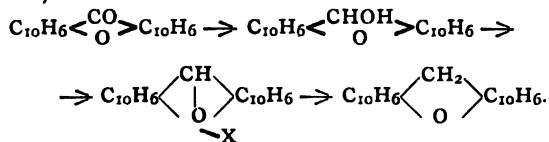
**A New Indicator: its Employment for the Research of Boric Acid in general, and particularly in Alimentary Substances.**—Lucien Robin.—The author prepares an indicator from the flowers of mimosa. It is of a pale yellow colour, but when a drop is added to 10 c.m. of distilled water no perceptible tint is observed. It behaves in the same manner as phenolphthalein, becoming bright yellow on the addition of alkalis and losing its colour when neutralised by acids. It is especially useful for titrating boric acid.

**Action of Magnesium and the Organo-magnesium Compounds on Bromophenetol.**—V. Grignard.—Under normal conditions the magnesium derivative of bromophenetol is not stable, nor are the analogous magnesium compounds derived from the ether oxides of arylaliphatic phenols. The author believes that the union of the aromatic organo-magnesium compounds—and especially the phenolic with bromophenetol, or with compounds of the same type as phenoxybromopropane-1-3, for example—will enable him to effect the synthesis of the primary halogen arylaliphatic compounds, whose preparation up to the present has been impossible when the lateral chain under consideration possesses more than an atom of carbon. He actually examines this generalisation.

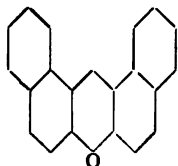
**Researches on the Dinaphthopyranic Series.**—R. Fosse.—The product of chromic oxidation of ethylidene di-β-naphthylene oxide does not appear to be a dinaphthopyrone. On contact with alkaline carbonates, the di-β-naphthyl carbonate decomposes into CO<sub>2</sub>, β-naphthol, and dinaphthopyrane, according to the equation



Starting with β-naphthyl carbonate, CO<sub>3</sub>(C<sub>10</sub>H<sub>7</sub>)<sub>2</sub>, the author obtains the following series of transformations:—Dinaphthopyrone (fuses 194°), dinaphthopyranol (fuses 145°), salts of dinaphthopyryl, dinaphthopyrane (fuses 201°).



All these bodies have the following molecular structure:—



**Oxytrotonic Lactone and the γ-Substituted Crotonic Acids.**—M. Lespieau.—By fixing two atoms of chlorine or bromine on to vinylacetic acid, CH<sub>2</sub>=CH—CH<sub>2</sub>—CO<sub>2</sub>H, the author obtains substituted

butyric acids in β and in γ, by the aid of which he prepares the γ-substituted crotonic acids.

*Bulletin de la Société Chimique de Paris.*  
Series 3, Vol. xxix., No. 15.

**The Existence of Arsenic in Bird's Eggs.**—Gabriel Bertrand.—As a result of his previous researches on the normal arsenic in the organism, the author has come to the conclusion that this metalloid—like carbon, sulphur, and phosphorus—is a constant element of the living cell, and that, instead of being localised in a few organs, it is, on the contrary, in all the tissues. If this is the case, arsenic should be found in the organism at all periods of life—in the embryonic cells as in the adult. It should be found therefore in bird's eggs, where the embryo is forced to develop without drawing on the outside world for any particle of arsenic it may need. For the purpose of testing this hypothesis the author has made three series of experiments. The first, in a measure preliminary, on ordinary eggs of commerce or "shop eggs"; the two others on eggs laid by hens kept specially under observation and carefully fed since the previous generation. In the end, arsenic was found in all the eggs used, both in the yolk, the white, the membrane, and the shells, in quantities varying from 5.0 to 0.5 thousandths of a milligram. per egg, according to the part of the egg examined. It is the yolk which is the richest in arsenic; it contains from one-half to two-thirds of the total amount present in the egg.

**The Decomposition of Carbonate of Lithium by Heat.**—P. Lebeau.—Already noticed.

**Commercial Silicated Manganeses.**—P. Lebeau.—Already noticed.

**Silicides of Chromium.**—P. Lebeau and J. Figueras.—Already inserted in full.

**New Method for the Qualitative and Quantitative Analysis of Osmides of Iridium.**—MM. Leidié and Quennessen.—Already inserted in full.

**New Method for the Estimation of Halogen Bodies in Organic Compounds.**—H. Baubigny and G. Chavanne.—Already noticed.

**New Method for the Estimation of Organic Matters in Waters, and particularly in those which contain Chlorides and Bromides.**—C. Lenormand.—Already inserted in full.

**Colloidal Silver.**—M. Hanriot.—Already noticed.

**The Benzoylised Derivatives of Hydrazobenzene and of its Homologues.**—P. Freundler.—Already noticed.

**The Sugar in the Milk of the Buffalo Cow.**—Ch. Porcher.—A controversial paper, in which the author disputes the statement made by Messrs. Pappel and Richmond, that the sugar in the milk of the buffalo cow is not lactose, but a special sugar to which they gave the name of tawfikose. He finds that this sugar answers to all the reactions of lactose.

**The Carbohydrates of Barley, and their Transformations during Germination.**—L. Lindet.—A long paper, not suitable for abstraction.

**Distribution of some Organic Substances in the Geranium.**—Eug. Charabot and G. Laloue.—The authors have found that in the geranium the volatile acids diminish as we pass from the leaf to the stalk; further, the terpenic compounds of the geranium are entirely localised in the leaf. Knowing now that these compounds are produced by the leaf and do not circulate through the stalks and stems, we can understand perfectly why the flowers of the geranium have no odour.

**A New Washer and a New Safety Tube.**—H. Vigreux.—This paper cannot be followed without the accompanying diagram.

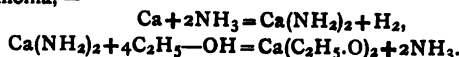
# MISCELLANEOUS.

**Petroleum Acts.**—It has recently come to our knowledge that attempts are being made by officers of local authorities to apply the legal test for petroleum to samples of liquid metal-polish, and we have satisfied ourselves by experiment that the results thus obtained are sometimes entirely misleading, as they do not represent the temperature of the portion of the liquid from which inflammable vapour is being evolved. In carrying out the test prescribed by the Petroleum Act, 1879, the sample under examination is slowly heated in a closed cup, and the temperature is indicated by a thermometer, the bulb of which is immersed in the liquid in the centre of the vessel. In these circumstances the heat communicated to the sample through the walls of the cup creates in such a liquid as petroleum convection currents, and through the circulation thus set up the temperature of the contents of the cup is equalised and the thermometer correctly indicates the temperature at which inflammable vapour is evolved by the liquid. On the other hand, if the sample contains solid matter in suspension, as is the case with the liquid metal-polish in question, the formation of convection currents is interfered with, and the surface of the liquid from which inflammable vapour is evolved, acquires a higher temperature than that of the portion in contact with the bulb of the thermometer. Thus, as we have ascertained experimentally, the thermometer may indicate a temperature of 59° F. when the temperature of the surface is 83° F., and a sample may be erroneously reported as having a flash-point below the legal limit of 73° F., when the true flash-point is far above the limit. No doubt this would have been provided for when the Act was passed if at that time the need for applying the test to such substances had been foreseen, but it was not until judgment in the case of the London County Council v. Holtzapfel's Compositions Company, Limited, was given in 1899 that mixtures containing petroleum were held to be petroleum within the meaning of the Acts. In our "Handbook on Petroleum," published in 1901, we referred on page 90 to the necessity for a stirrer in the oil cup when the test specified in the Petroleum Act is employed for the testing of paints and other substances containing petroleum, and when opportunity occurs for a revision of the law this addition will doubtless be legalised. We are, however, of opinion that in the meantime authorities charged with the administration of the Petroleum Acts should be made aware of the circumstances we have referred to, and that testing officers should take steps to ascertain the true flash-point of samples of liquid metal-polish or other substances which are not thoroughly liquid, and therefore cannot be satisfactorily tested in precise accordance with the directions given in the schedule to the Act. In many instances it may be possible to obtain a sample of the petroleum used in the substance, or the solid matter present in the sample can be removed by straining or filtration, care being taken to avoid loss of the more volatile constituents by evaporation, when the separated liquid can be tested in the prescribed manner. The liquid should not, however, be separated by distillation, as this operation may yield a distillate of lower flash-point than that of the petroleum with which the mixture was made. For guidance in determining whether there has been any infraction of the law, the sample may also be tested in an apparatus provided with an efficient stirrer. In any case of doubt as to the true flash-point of the material we would suggest that reference should be made to H.M. Inspectors of Explosives at the Home Office, who will be prepared to give advice as to the course which should be adopted.—(Signed) J. H. THOMSON, Captain, H.M. Chief Inspector of Explosives; BOVERTON REDWOOD, Adviser on Petroleum to the Home Office; April 30th, 1904.

**Silicic Acid (II).**—E. Jordis and E. H. Kanter.—The preparation of a pure colloidal solution of silica is difficult.

Contrary to what has been stated by Graham, the authors have only been able to obtain very dilute solutions by his method. The proportion, 12 per cent, given by him (*Comptes Rendus*, vol. lix., p. 174), appears to be mistaken, for as soon as the amount reaches 1·8 to 2·0 per cent a deposit of gelatinous silica is formed. This figure is close to that obtained by Grimaux (*Comptes Rendus*, vol. xcvi., p. 1485), who succeeded in obtaining a solution of silica containing 2·26 per cent, by the decomposition of methylic ether,  $\text{Si}(\text{OCH}_3)_4$ , by boiling water. By boiling gelatinous silica with water in a flask with a vertical condenser, we obtain solutions, the strength of which may reach 0·15 per cent, but it appears that this relatively high percentage is only due to traces of foreign matters, as the same silica boiled afresh only goes into solution in much slighter proportions (0·047 to 0·027 per cent). Grimaux's figure is too high for the same reasons. On repeating his experiments the proportion of silica was lowered to 0·1 per cent, and, further, the silica obtained by evaporation and calcination was blackened on account of the presence of organic matter. If, during the preparation by means of dialysis, we add a drop of acid or alkali, the proportion of silica increases to 10 per cent. In conclusion, silica does not give a pure hydrosol, but must be looked upon as an amphoteric substance.—*Zeit. Anorg. Chem.*, vol. xxxv., p. 16.

**Action of Calcium on Alcoholic Ammonia.**—G. Doby.—Ethylate of calcium, which has not yet been prepared in a pure state, takes rise in the reciprocal action of ammonia gas, calcium, and ethylic alcohol. Amidide of calcium is first formed, and this is then transformed by the alcohol into ethylate with the regeneration of the ammonia,—



Ethylate of calcium crystallises with 2 molecules of alcohol, which can be eliminated at about 50°. At the same time as the ethylate,  $\text{Ca}(\text{C}_2\text{H}_5\text{O})_2 + 2\text{C}_2\text{H}_5(\text{OH})$ , is formed, the action of the ammonia and the alcohol on the calcium gives another compound in hexagonal crystals having the formula  $\text{CaO}_3\text{C}_2\text{H}_6\text{O}$ . These crystals are apparently identical with those obtained by De Porcand (*Comptes Rendus*, vol. cxix., p. 1266), and are only formed on account of the presence of a little moisture. Ethylate of calcium occurs in white needles, soluble in hot alcohol; the solution becomes brown on contact with the atmospheric oxygen; when exposed freely to the air it gives the carbonate. On contact with water, the anhydrous ethylate is decomposed into  $\text{Ca}(\text{OH})_2$  and alcohol. The same ethylate is also formed by the action of hydride of calcium upon alcohol, according to the equation,  $4\text{C}_2\text{H}_5\text{—OH} + \text{CaH}_2 = (\text{C}_2\text{H}_5\text{O})_2\text{Ca} \cdot 2(\text{C}_2\text{H}_5\text{—OH}) + 2\text{H}_2$ .—*Zeit. Anorg. Chem.*, vol. xxxv., p. 93.

**Suboxide of Phosphorus, and the Solubility of Red Phosphorus in Alcoholic Aqueous Potash.**—A. Michaelis and K. Von Arend.—After having announced that the suboxide of phosphorus of Michaelis and Pitsch was impure amorphous phosphorus, D. L. Chapman and F. A. Lidbury confirmed the existence of this suboxide. They announced, further, the curious fact that commercial red phosphorus was soluble in alcoholic aqueous potash. Their conclusions on this point are as follows:—1. Red phosphorus, as well as ordinary crystalline phosphorus, and the amorphous phosphorus obtained by heating phosphorous acid with trichloride of phosphorus, is insoluble in alcoholic aqueous alkali. 2. If red phosphorus is ground for a long time with water, it becomes partially oxidised to the state of suboxide, and to a very slight extent to the state of acids of phosphorus. 3. In the absence of water (toluene, sulphide of carbon) a portion of the ground-up phosphorus becomes transformed into ordinary phosphorus which, as we know, is soluble in the alkalis.—*Liebig's Annalen*, vol. cccxxxv., p. 361.

## NOTES AND QUERIES.

\* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

**Colouring Varnish.**—I have recently been trying to colour an essential oil (rosemary) varnish with alizarine—red, brown, and purple. For the red I used sodium carbonate and alum; for the brown, oxide of iron; and for the purple, caustic potash. I am highly pleased with these colours, but I find that the salts form an emulsion with, or saponify, the gums and precipitate them, but not the alizarine. Therefore what I want to find out is how to get these alizarine or madder colours (which are practically the same) into the varnish without at the same time emulsifying or precipitating the gums.—CHAS. E. POLLARD, Cape Town.

## MEETINGS FOR THE WEEK.

**TUESDAY, 31st.**—Royal Institution, 5. "The Solar Corona," by H. F. Newall, F.R.S., &c.  
Society of Arts, 4.30. "The Economic and Industrial Progress and Condition of India," by J. E. O'Connor, C.I.E.

**WEDNESDAY, June 1st.**—Society of Public Analysts, 8. "The Analysis of Condensed Milk," by J. B. P. Harrison. "Roasted Beetroot," by E. G. Clayton. "A Collection of Readings with the Zeiss Oleo-butyrometer," by William Chattaway and C. G. Moor. "Note on the Estimation of Sugars and Starch in Vegetable Substances," by John S. Ford.

**THURSDAY, 2nd.**—Royal Institution, 5. "Literature and the State," by H. G. Wells, B.Sc.

Chemical, 8. "Izo-Nitrosocamphor," by M. O. Forster. "Imino-ethers and Allied Compounds corresponding with the Substituted Oxamic Esters," by G. D. Lander. "Action of Heat on  $\alpha$ -Hydroxycarboxylic Acids—Part I.,  $\alpha$ -Hydroxystearic Acid," by H. R. Le Sueur. "The Basic Properties of Oxygen—Additive Derivatives of the Halogen Acids and Organic Compounds and the Higher Valencies of Oxygen—Asymmetric Oxygen," by E. H. Archibald and D. McIntosh.

**FRIDAY, 3rd.**—Royal Institution, 9. "The Development of the Theory of Electrolytic Dissociation," by Prof. Svante Arrhenius.

**SATURDAY, 4th.**—Royal Institution, 3. "Spitsbergen in the Seventeenth Century," by Sir William Martin Conway.

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### CONTENTS.

| ARTICLES:—                                                                                                                         | PAGE |
|------------------------------------------------------------------------------------------------------------------------------------|------|
| The Boiling-points of Homologous Compounds, by Hugh Ramage, B.A. ....                                                              | 265  |
| Further Experiments on the Production of Helium from Radium, by Sir William Ramsay, K.C.B., F.R.S., and Frederick Soddy, M.A. .... | 266  |
| On the Kmanation Substance (Emanium), by F. Giesel ....                                                                            | 267  |
| Colorimetric Estimation of Chromium, by A. Moulle ....                                                                             | 268  |
| The Action of Copper on Chloric Acid, with and without the Assistance of Electrolysis, by André Brochet ....                       | 269  |
| Radio-active Minerals and Substances. ....                                                                                         | 270  |
| PROCEEDINGS OF SOCIETIES—                                                                                                          |      |
| CHEMICAL SOCIETY ....                                                                                                              | 271  |
| NOTICES OF BOOKS ....                                                                                                              | 274  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES ....                                                                                         | 274  |
| MISCELLANEOUS ....                                                                                                                 | 275  |
| MEETINGS FOR THE WEEK ....                                                                                                         | 275  |

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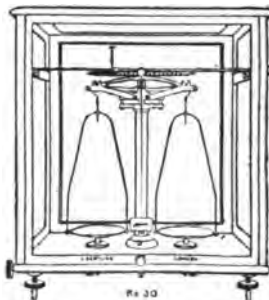
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2323.

## THE BOILING-POINTS OF HOMOLOGOUS COMPOUNDS.

By HUGH RAMAGE, B.A., St. John's College.

THE relations between certain properties of elements and their atomic masses have been described by the writer in *Roy. Soc. Proc.*, 1902, lxx., pp. 1 and 303. These relations were discovered by a study of diagrams drawn with the physical constants as abscissæ and the atomic masses—or the squares of these—as ordinates. This graphical method does not appear to have been employed by other workers, except in the simplest form, for showing that the properties are *periodic* functions of the atomic masses.

The method has been extended to other properties of the elements, and also to those of compounds. Amongst the latter, the boiling-points offer the most complete data, and in addition furnish constants which are amongst the least affected by other factors—such, for instance, as association of the molecules,—and, moreover, they are, especially amongst organic compounds, the most accurately determined of the physical constants. A study of the boiling-points of many substances has been made, and some facts relating to the boiling-points of homologous compounds will be dealt with in this paper.

Professor James Walker has given a general formula,  $T = aM^b$ , for the boiling-points of homologous compounds (*Trans. Chem. Soc.*, 1894, lxx., 193, 725), in which  $T$  is the boiling-point in absolute degrees,  $M$  is the molecular weight, and  $a$  and  $b$  are constants for each series. He applied it to several series, and amongst them to the normal paraffins from  $C_7H_{16}$  to  $C_{16}H_{34}$ . He found that it did not apply to the alkyl bromides and iodides, the normal alkylamines, and the normal ketones and aldehydes. He also stated that no formula of this type could possibly be applied to the alcohols.

It was observed when drawing the curve through the boiling-points of the paraffins that it would, if produced below methane, pass very near to the position of hydrogen; it seemed at first, when using the older figure for methane (113°), that hydrogen might be regarded as a member of the series. It was, however, found by drawing a diagram with the logarithms instead of the numbers, that hydrogen did not fall on the curve, but it fell near it. The change from  $H_2$  to  $CH_4$  is not very different from what it would be were hydrogen the first member of the series.

In referring to the ten hydrocarbons to which his formula applies, Walker remarked:—"In this particular case the formula is unusually simple, for the constant  $b$  becomes equal to 0.5, the curve of boiling-points being therefore a true parabola." He does not discuss the point further. The curve to Walker's formula was drawn on the diagram, and in studying the two curves it appeared probable that the simple formula only applied exactly to the  $CH_2$  chain linkage, and that the influence of the terminal hydrogen atoms was either a constant in the higher members of the series, or it was so small that it might be disregarded. The influence of these terminal atoms increases as the chain shortens and the atoms approach each other until, in methane, the diagram indicates we practically have the effect of two  $CH_2$  groups. This work has suggested a modification of the formula which applies to the series from methane to hexadecane.

The new formula is  $T = a \{M(1 - 2^{-n})\}^{\frac{1}{2}}$ ; where  $n$  is the number of carbon atoms in the molecule. The constant  $a$  is the same as Walker used. It may, however, be expressed as a function of the pressure, and it is given approximately for pressures of 15, 30, 50, 100, and 760 m.m. by the equa-

TABLE I.

| Paraffin.         | Observed boiling-point. | Calculated boiling-point. | Differences. |
|-------------------|-------------------------|---------------------------|--------------|
| $CH_4$ ..         | 108.3                   | 105.7                     | -2.6         |
| $C_2H_6$ ..       | 180.0                   | 177.3                     | -2.7         |
| $C_3H_8$ ..       | 228.0                   | 231.9                     | +3.9         |
| $C_4H_{10}$ ..    | 274.0                   | 275.6                     | +1.6         |
| $C_5H_{12}$ ..    | 309.3                   | 312.2                     | +2.9         |
| $C_6H_{14}$ ..    | 342.0                   | 343.9                     | +1.9         |
| $C_7H_{16}$ ..    | 371.4                   | 372.3                     | +0.9         |
| $C_8H_{18}$ ..    | 398.5                   | 398.3                     | -0.5         |
| $C_9H_{20}$ ..    | 422.5                   | 422.5                     | 0            |
| $C_{10}H_{22}$ .. | 446.0                   | 445.2                     | -0.8         |
| $C_{11}H_{24}$ .. | 467.5                   | 466.8                     | -0.7         |
| $C_{12}H_{26}$ .. | 487.5                   | 487.3                     | -0.2         |
| $C_{13}H_{28}$ .. | 507.0                   | 507.0                     | 0            |
| $C_{14}H_{30}$ .. | 525.5                   | 526.0                     | +0.5         |
| $C_{15}H_{32}$ .. | 543.5                   | 544.2                     | +0.7         |
| $C_{16}H_{34}$ .. | 560.5                   | 561.9                     | +1.4         |

TABLE II.

| Alcohol.  | Observed boiling-point. | Calculated boiling-point. | Differences. |
|-----------|-------------------------|---------------------------|--------------|
| Methyl .. | 337.7                   | 331.1                     | -6.6         |
| Ethyl ..  | 351.3                   | 351.1                     | -0.2         |
| Propyl .. | 370.4                   | 370.8                     | +0.4         |
| Butyl ..  | 390.0                   | 390.5                     | +0.5         |
| Amyl ..   | 411.0                   | 410.3                     | -0.7         |
| Hexyl ..  | 430.0                   | 430.0                     | 0            |
| Heptyl .. | 449.0                   | 449.8                     | +0.8         |
| Octyl ..  | 469.0                   | 469.4                     | +0.4         |

TABLE III.

| Aldehyde.        | Observed boiling-point. | Calculated boiling-point. | Differences. |
|------------------|-------------------------|---------------------------|--------------|
| Formic ..        | 252.0                   | 267.0                     | +15          |
| Acetic ..        | 293.8                   | 294.0                     | +0.2         |
| Propylic ..      | 321.8                   | 321.0                     | -0.8         |
| Butylic ..       | 348.0                   | 348.0                     | 0            |
| Amylic ..        | 376.4                   | 375.0                     | -1.4         |
| Capric ..        | 401.0                   | 402.0                     | +1.0         |
| Ketone.          |                         |                           |              |
| Acetone ..       | 330.0                   | 335.0                     | +5           |
| Di-ethyl ..      | 376.0                   | 376.0                     | 0            |
| Di-propyl ..     | 417.0                   | 417.0                     | 0            |
| Di-butyl ..      | —                       | 458.0                     | —            |
| Di-amyl ..       | 499.0                   | 499.0                     | 0            |
| Di-hexyl ..      | 536.0                   | 540.0                     | +4           |
| Methyl-ethyl ..  | 354.0                   | 355.5                     | +1.5         |
| Methyl-propyl .. | 375.0                   | 376.0                     | +1           |

tion  $a = 23.5 P^{0.07}$ ; where  $P$  is the pressure. Calculated from the first sixteen members of the series for a pressure of 760 m.m., its mean value is 37.3775—a figure which is practically identical with that calculated by the simpler formula (37.38) from the higher members of the series. When  $n = 11$  and upwards the two formulæ give the same results to the first decimal place. Table I. gives the observed and calculated numbers and the differences between them.

The simple formula gives a difference of +41.2 for methane and of +24.7, 19.9, 10.6, 7.9, 4.6, and 2.4 respectively for the succeeding members of the series.

### Normal Alcohols.

The line which passes through the points given by the molecular weights and boiling-points of water and the alcohols, is sharply curved at the beginning, and then it becomes almost straight. The following linear equation gives the boiling-points of the alcohols:—

$$T = 286.2 + 1.41 M.$$

Table II. gives the observed and calculated numbers in absolute degrees.

*Aldehydes and Ketones.*

The boiling-points of these are also given by linear equations—

1. Aldehydes . . .  $T = 209.14 + 1.9286 M.$
2. Ketones . . .  $T = 250.07 + 1.4643 M.$

The observed and calculated numbers, in absolute degrees, are given in Table III.

It is worthy of remark that the difference in boiling-points of two ketones differing by  $\text{CH}_2$  is about  $41.0^\circ$ , and this is the difference between the first constants in the above equations.

The influence of the oxygen atoms in the compounds given in the last two tables is very marked, as it also is in the acids. The curve representing the acids is irregular, and it indicates that the boiling-points of the lower members are abnormal, due doubtless to the association of their molecules, whilst the boiling-points assume a normal value in the higher members of the series.—*Proceedings of the Cambridge Philosophical Society*, vol. xii., Part 5.

### FURTHER EXPERIMENTS ON THE PRODUCTION OF HELIUM FROM RADIUM.\*

By Sir WILLIAM RAMSAY, K.C.B., F.R.S.,  
and  
FREDERICK SODDY, M.A.

(Concluded from p 258).

*Experiment 5.*—The former experiment was repeated, this time with a regular capillary tube, of which the volume per centimetre was  $0.24$  cub. m.m. It was regular in bore, and the depression due to capillarity was  $56.2$  m.m. of mercury. It was heated, as well as the bulb in which the emanation was to be condensed, to incipient redness during evacuation. The emanation was introduced, the accompanying hydrogen pumped off, and the liquid air jacket removed. The volume of the emanation was read at once, at different pressures. The following table gives the lengths of the capillary tube, the corresponding volumes, the pressures corrected for capillarity, and the products of volume into pressure:—

| Length of tube. | Volume in cub. m.m. | Pressure in m.m. | Volume $\times$ pressure. |
|-----------------|---------------------|------------------|---------------------------|
| 6.80            | 0.163               | 132.4            | 21.6                      |
| 2.30            | 0.0552              | 333.4            | 18.4                      |
| 1.55            | 0.0372              | 518.1            | 19.3                      |
| 1.20            | 0.0288              | 644.8            | 18.6                      |
| 0.95            | 0.0228              | 765.8            | 17.5                      |
| 2.55            | 0.0612              | 309.2            | 18.9                      |
| 11.90           | 0.372               | 55.3             | 20.6                      |

The mean value of the product is  $19.3$ , and the volume at normal pressure  $0.0254$  cub. m.m. The same afternoon, numerous readings were taken, and it was found that the sticking of the mercury in the capillary tube made it difficult to ascertain the true volume. As the pressure, however, was first raised and then lowered, the mean cannot be far from the truth. Now, a most remarkable circumstance must be chronicled. Whereas the emanation in the previous experiment contracted during its whole life, in this experiment a regular expansion was observed, rapid at first, and slowly falling off from day to day. Between five minutes past 1 o'clock and 7 o'clock, the pressure being kept constant at  $55.3$  m.m., the value of P.V. increased from  $20.6$  to  $48.4$ . This was on January 20th. On the 21st, the P.V. had increased to  $71.2$ , and remained fairly constant all day, and three small bubbles appeared in the thread of mercury, below the level of the emanation. On the 22nd, the value of P.V. had diminished to  $56.5$ , and the volume of the bubbles had increased to  $2.7$  m.m. length in the tube. On the 23rd, the emanation occupied

practically the same volume, but the length of the bubbles had increased to  $4.1$  m.m. The presence of these bubbles made it impossible to obtain correct readings, for the "sticktion" of the mercury was much increased. On the 25th, P.V. had further diminished to  $51.2$ , and the length occupied by bubbles had increased to  $5.5$  m.m. On February 3rd, the bubbles were united with the emanation; the value of P.V. was  $132.5$  and the volume of the gas under normal pressure,  $0.174$  cub. m.m. On the 9th, the P.V. had increased to  $166$ , and the volume at normal pressure to  $0.224$  cub. m.m. Lastly, the volume of the gas was measured on the 12th at atmospheric pressure; it amounted to  $0.262$  cub. m.m. The level of the mercury was then lowered, and the gas pumped out; it showed a brilliant spectrum of helium. The tube was then heated, and the volume of the absorbed gas was  $0.103$  cub. m.m. at atmospheric pressure; it, too, showed the helium spectrum, but the tube punctured before this could be confirmed.

These results are somewhat inexplicable in the light of the former experiment. More stringent precautions had been taken to free the capillary tube from gas in the second experiment than in the first, and yet bubbles appeared below the surface of the mercury. It may be that owing to the quality of the glass of which the first tube was made, the helium found means to enter into its substance more easily than into that of the second. But at any rate, the volume produced is of the same order, as the following considerations will show.

On the view that the emanation results from a definite fraction of the radium disintegrating per second, this fraction can be calculated from the volume of the emanation, and the time of accumulation. The emanation accumulates until the rate of production is balanced by the rate of disappearance, and then the quantity remains constant. Let  $Q_\infty$  be the equilibrium quantity, and  $Q_t$  the quantity present after time  $t$ ,—

$$Q_t / Q_\infty = 1 - e^{-\lambda t},$$

where  $t$  is expressed in seconds, and  $\lambda$  is a constant representing the proportion of the emanation changing per second, and equals  $1/463,000$  (Rutherford and Soddy, *Phil. Mag.*, 1903, [6], vol. v., pp. 445 and 576). The radium bromide employed weighed about 60 milligrams. Assuming that the compound contained about half its weight of the element (radium, 225; bromine +  $2\text{H}_2\text{O}$ , 196), the quantity of radium may be taken as about  $0.03$  gm. In the first experiment, the time of accumulation  $t$  was eight days =  $691,200$  seconds;  $Q_t$  therefore equals  $0.775 Q_\infty$ . The volume taken ( $0.027$  cub. m.m.) in the first experiment was that at the end of the first day, and a correction must be applied to allow for the amount that had changed in this interval. The quantity remaining after the lapse of one day is  $0.83$  of the initial quantity. The volume,  $0.027$  cub. m.m., is therefore—

$$0.83 \times 0.775 Q_\infty = 0.643 Q_\infty.$$

The average life of the particle in a system in which a constant fraction  $\lambda$  of the number of particles changes per second can be shown to be  $1/\lambda$ . The equilibrium quantity,  $Q_\infty$ , is the quantity produced in the period of average life of the atom of the emanation, or  $Q_\infty = Q_0/\lambda = 463,000 Q_0$ , where  $Q_0$  is the quantity produced per second. And  $0.643 Q_\infty = 297,830 Q_0$ . The volume of  $Q_0$  is thus  $0.027/297,830 = 9.1 \times 10^{-7}$  cub. m.m. This is in the case of  $0.03$  gm. of radium; 1 gm. of radium, therefore, produces  $3 \times 10^{-6}$  cub. m.m. of emanation per second.

Since the emanation resembles the gases of the argon family in chemical inertness, its molecule is probably monatomic, and its atomic weight must be twice its density in terms of hydrogen as unity. The density is not accurately known; but diffusion experiments indicate a value of about 80. The atomic weight being therefore in the neighbourhood of 160, not more than one atom of emanation can be produced from one atom of radium. To

\* A Paper read before the Royal Society, April 28, 1904.

determine the ratio between the quantity of emanation and the quantity of radium producing it, it is necessary to know the volume that would be occupied by the radium in the form of monatomic gas. This is for 1 grm. of radium  $(2 \cdot 11 \cdot 2) / 225 = 0 \cdot 01$  litre =  $10^4$  cub. m.m. One grm. of radium produces  $3 \times 10^{-6}$  cub. m.m. of emanation per second, and if one atom of radium produces one atom of emanation, then  $\lambda$ , the proportion of the radium changing per second, is  $3 \times 10^{-11}$ . The proportion changing per year is  $9 \cdot 5 \times 10^{-4}$ ; thus slightly less than one-thousandth part changes per year. The average life of the radium atom is  $1/\lambda = 3 \cdot 3 \times 10^{10}$  seconds = 1050 years.

In the second experiment, the emanation was accumulated six days, and measured 0.0254 cub. m.m. In this case,—

$$Q_t = 0 \cdot 674 Q_{\infty} = 312,060 Q_0,$$

and  $Q_0 = 0 \cdot 81 \times 10^{-7}$  cub. m.m.;  $\lambda = 2 \cdot 4 \times 10^{-11}$ , and  $1/\lambda = 1250$  years. The mean of the two experiments, therefore, gives for 1 grm. of radium (element)  $Q_0 = 2 \cdot 85 \times 10^{-8}$  cub. m.m.;  $Q_{\infty} = 1 \cdot 3$  cub. m.m.,  $\lambda = 2 \cdot 85 \times 10^{-11}$ , and  $1/\lambda = 1150$  years.

Rutherford and Barnes (*Phil. Mag.*, 1904, [6], vol. vii., p. 202) have shown that 75 per cent. of the total heat-evolution of radium which has reached its equilibrium state is derived from the emanation and its subsequent products of change. Since 1 grm. of radium evolves 100 calories per hour (Curie), 1.3 cub. m.m. of emanation emit 75 calories per hour. The total quantity of heat  $H$  emitted during the complete change is given by multiplying  $h$  the emission per second, by the average life of the emanation in seconds, thus giving  $H = h/\lambda = 9646$  calories. One c.c. of emanation would therefore emit  $7 \cdot 4 \times 10^6$  calories during its complete change. A c.c. of hydrogen and oxygen in the proportion required to form water evolve 2.04 calories on explosion, or a quantity 3,600,000 times less than is emitted by an equal volume of the radium emanation. If the density of the emanation is assumed to be 100, the ratio of the energies emitted by equal weights of emanation and of water is 216,000 to 1.

The total quantity of energy evolved during the change of 1 grm. of radium is given by multiplying the energy emission per second by the average life of the radium atom in seconds, and is  $10^6$  calories. The energy evolved in the formation of 1 grm. of water is  $3 \cdot 8 \times 10^6$  calories; hence the ratio is again about 250,000 to 1.

The volume of  $Q_{\infty}$ , the equilibrium quantity of emanation produced by 1 grm. of radium, was theoretically calculated by Rutherford (*Nature*, August 20, 1903) from the energy emitted by radium per second, and the energy of the  $\alpha$ -particle, as calculated from its mass and its velocity. By making certain assumptions about to be considered, he deduced that  $Q_{\infty}$  must lie between 0.6 and 0.06 cub. m.m. The maximum estimate, which is almost exactly one-half the experimental value found by us, was based on the assumption that the whole, and the minimum estimate on the assumption that only one-tenth, of the energy of disintegration is manifested in the kinetic energy of the  $\alpha$ -particle expelled. It was further assumed that only one  $\alpha$ -particle was expelled from each atom, at each disintegration accompanied by  $\alpha$ -radiation that is known to occur. If more than one  $\alpha$ -particle is expelled at each disintegration, the theoretical estimate must be correspondingly reduced. Since the experimental value exceeds the maximum theoretical estimate, it follows that there are now direct experimental reasons for believing that—

1. Only one  $\alpha$ -particle is expelled from the atom at each disintegration.
2. The greater part of the energy of disintegration appears in the form of kinetic energy of  $\alpha$ -radiation.
3. The emanation is a monatomic gas.

It must be remembered that the experimental value is necessarily a maximum value; for if any impurity were present with the emanation, it would increase the volume measured.

## ON THE EMANATION SUBSTANCE (EMANIUM).\*

By F. GIESEL.

THE investigation of the spark spectrum of the emanation substance, which Runge and Precht very kindly undertook, has proved that the substance consists essentially of lanthanum with a little cerium. Thorium, barium, and radium were not present. New lines could not be observed, it being impossible to say anything definite on this point owing to the enormous number of lines.

The anhydrous chloride or bromide (to a less degree the sulphate) which phosphoresces spontaneously, show a discontinuous phosphorescence spectrum, consisting of three lines, from about the red to the blue-green, at approximately equal distances.† The determination of the position of the lines is made much more difficult owing to the feebleness of the light, but the determination must be made by the photographic method in order to see whether the spectrum of a new rare earth is obtained.

During a further period of observation lasting one year the following results relating to the properties of the "emanation substance" have been obtained.

Glass vessels in which the substance has been kept for some months become violet coloured up to the level to which they were filled. Paper becomes brown, and is destroyed.

First of all I convinced myself that the activity of the solid salts suffers absolutely no further change as soon as the maximum has been reached, which occurs after about one month after they have separated out from the solution. The conditions are exactly similar to those which I have described for radium (*Ann. d. Phys. u. Chem.*, 1899, lxi., 92). The original ( $\beta$ -) radiation is smaller the longer the substance is kept in solution and the more dilute it is.

This behaviour characteristic of the primarily active elements thus removed the last doubt in my mind as to whether the "emanation substance" contains a new radioactive element (rare earth) different from radium. Thus it is not possible to impart artificially the characteristic power of emanation to inactive rare earths by contact with radium. If solutions of thorium, lanthanum, cerium, &c., to which radium has been added are precipitated with ammonia and washed, the precipitates are adulterated with traces of radium, and show, besides slight  $\beta$ -radiation, remarkably strong  $\alpha$ -radiation, but yet no emanation similar to the emanation substance.

It has further appeared that all emanating substances which do not belong to the rare earths, such as the insoluble residue (*Berichte*, 1903, xxxvi., 343), which has not been further examined, the magnesium, strontium (and barium) precipitates lose their activity and power of emanation gradually, even if very slowly. These substances have originated secondarily by the influence of the emanation substance (by induction); unlike the rare earths they possess the maximum of activity and emanation immediately after separation; the gradual disappearance may require a year or longer.

We must not forget to notice that the complete removal of the emanation substance, especially in working with small quantities, as may be supposed, presents greater difficulties than that of radium. For example, with a magnesia preparation the activity was only very little diminished after one year. It may be assumed that the magnesia still contains traces of the emanation substance, even if no further precipitate is obtained with oxalic acid. It is just as difficult to free uranium oxide from the

\* *Berichte*, 1903, xxxvi., 342.

† I have never been able to observe a discontinuous spectrum in self luminous radium bromide melted in glass tubes under ordinary pressure with air or hydrogen. The radium bromide in an atmosphere of hydrogen does not become brown as in air, but blue-black. The peculiar scintillating phosphorescence can in this case be produced again by heating for a short time (with decolouration) as often as is required without becoming fainter, probably because the altered bromide can be completely regenerated. (See *Berichte*, 1902, xxxv., 3609).



emanation substance if it occurs as an adulteration of this substance, so that it does not retain a considerable activity. Uranium potassium sulphate prepared from it is strongly self-luminous. The small activity of the commercial uranium salts seems to me to depend rather on traces of the emanation substances than on radium if the uranium itself is inactive. The fact that in spite of previous repeated purification of the commercial uranium nitrate, the subsequent fractions exhibit not the same but different activity is in agreement with this theory.

Small quantities of lead, if they still adhere accidentally to the emanation substance and are afterwards separated by sulphuretted hydrogen, are very active, and remain so. I think that my former strongly active lead preparations (*Berichte*, 1902, xxxv., 102) have borrowed their activity from the emanation substance, especially as they were always found only in its immediate neighbourhood.

The feebly active lead salts of pitchblende (I have not yet been able to obtain the lead from pitchblende in a perfectly inactive state) which show an increase of activity, can also be infected by the most minute traces of radium.\*

If an ordinary barium salt is added artificially to a solution of a feebly active rare earth and the barium is precipitated with sulphuric acid, the barium sulphate is more strongly active than the rare earth. By fractional crystallisation of the purified barium bromide the activity may be very quickly increased, just as in the case of the barium-radium mixture. The anhydrous salt phosphoresces strongly. Runge and Precht have compared the spark spectrum with equally active radium barium bromide. In the latter preparation the principal radium lines could be easily seen, but in the former they were not present. The most convenient method of distinguishing the two preparations is afforded by the immediate appearance of the maximum ( $\lambda$ ) activity, and by the power of emanation which may be shown by means of the zinc sulphide screen. Debiere (*Comptes Rendus*, cxxxi., 333) has previously obtained similar results by precipitation of barium sulphate from thorium-actinium solutions and crystallisation of the chloride. Unfortunately I have as yet had no opportunity of arranging comparative experiments with thorium from pitchblende and my lanthanum preparations, which would be of special importance in relation to the emanation.

The induced activity produced by the emanation in air is different as regards its disappearance from that in aqueous solution. Radium behaves similarly. But the inducing force is greater than in the case of the radium emanation, as I have specially noted before. Thus, for example, wadding, through which the emanation has been led for a short time, becomes as active as the substance itself; the disappearance only requires some hours. Goldstein (*Verh. d. Dtsch. Phys. Ges.*, November 13, 1903) has also recently observed the easy and rapid transposition of my emanation into induced activity. I have not been able to obtain an absorption (recognisable on the phosphorescence screen) by means of liquids.

I shall now endeavour—from the best lanthanum preparations thus accumulated—to isolate the new active rare earth. The effect of the preparations is very astonishing. It appears as a splendid phenomenon, visible to a great distance in a large lecture room, if a current of air is blown through tubes against a Sidot's blende screen of about  $\frac{1}{4}$  sq. m. surface over the preparations, enclosed in paper capsules, and collected in a glass flask. The scintillation of the screen, which lasts for some time, is visible to the naked eye if the screen is held before the eye beyond the normal distance of sight. The sparks are clearer and larger respectively than with radium or polonium, hence a spinthariscopes prepared with the emanation substance is far more effective. I have also been able to see the scintillation of ordinary glass in the flask in which it was kept (besides the uniform phosphorescence); but it appears much less striking than with Sidot's blende.

I shall for the future call the strongly radio-active

element, which is to be assumed as present in the emanation substance, and is presumably allied to lanthanum, "Emanium."—*Berichte der Deutsch. Chem. Ges.*, 1904, xxxvii., 1696.

## COLORIMETRIC ESTIMATION OF CHROMIUM.

By A. MOULIN.

M. CAZENEUVE (*Bull. Soc. Chim.*, vol. xxix., p. 758) has already described the coloured reaction produced when diphenylcarbazine is brought into contact with chromic acid, or the chromates in general. A very intense purple-violet colour is thus produced; the reaction is extremely sensitive, and will show traces of chromium. As it only requires a relatively short time to acquire its final intensity, which then remains for some time, it seemed to us that it might very well be used for the rapid estimation of this metal.

Two solutions are necessary for this estimation:—(1) A solution of diphenylcarbazine; (2) A titrated solution of chromic acid.

1. *Solution of Diphenylcarbazine*.—The solution of diphenylcarbazine is prepared in the following manner:—

|                   |    |    |    |     |       |
|-------------------|----|----|----|-----|-------|
| Diphenylcarbazine | .. | .. | .. | 2   | grms. |
| Alcohol at 90°    | .. | .. | .. | 100 | c.c.  |
| Acetic acid       | .. | .. | .. | 10  | "     |

Boil on the water-bath until completely dissolved, and make the volume up to 200 c.c. with alcohol at 90°.

2. *Chromic Solution*.—Weigh out 0.5 gm. of pure chromic acid, dissolve it in a little water, and make up the volume to 1000 c.c.; take 100 c.c. of this solution, place them in a graduated litre flask, and make up to 1000 c.c. with distilled water; thus we have a solution of which each c.c. represents 0.00005 of chromic acid, or 0.000026 of chromium.

We then weigh out 0.25 to 0.50 gm. of the sample to be examined, according to the supposed proportion of chromium present, dissolve it, and transform the chromium salt obtained into chromate by one of the ordinary methods.

With chrome-irons we obtained very good results by oxidising the salt of chromium, at boiling temperature, by peroxide of hydrogen in the presence of an excess of potash. This method of oxidation by peroxide of hydrogen has given us excellent results in every case.

When the oxidation is complete, we filter to separate the metals precipitated by the potash, wash the filter, and make the volume up to 100 c.c. or 200 c.c. according to circumstances, after having neutralised exactly with acetic acid. We then pour into graduated test glasses of 100 c.c. capacity, 2 c.c. of the diphenylcarbazine solution, and add about 70 c.c. of distilled water; into these test glasses we measured off 0.5 c.c., 1 c.c., 1.5 c.c., and 2 c.c., &c. of the titrated chromic solution, and into corresponding test glasses a known number of c.c. of the solution to be estimated; make up the volumes to 100 c.c., and after mixing leave them for twenty minutes for the reaction to become complete.

We then make the tints of the two series equal, and from this we can deduce the amount of chromium in the sample under examination.

This last operation can be simplified by the use of a Dubosq colorimeter.

This process, which we have often used, has given us very exact results, but it is essential, if the results are to be perfectly comparable, that the dilution of the chromate in the solution under examination should be as near as possible to that of the titrated solution because of the extreme sensitiveness of the reaction. It is also always preferable to add an excess of diphenylcarbazine solution; in fact, if the amount of the reagent used is insufficient the reaction becomes modified completely, and the general colour becomes more reddish, making the comparison with the scale difficult.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 6.

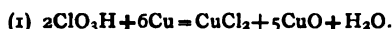
\* Lead nitrate, for example, crystallises with barium radium nitrate.

THE ACTION OF COPPER ON CHLORIC ACID  
WITH AND WITHOUT THE ASSISTANCE  
OF ELECTROLYSIS.

By ANDRÉ BROCHET.

CHLORIC acid attacks copper more or less energetically according to its concentration. With a solution containing 280 grms. of  $\text{ClO}_3\text{H}$  (density = 1.15) per litre, the attack at about  $60-80^\circ$  is tumultuous; chlorised fumes are given off, but the reaction becomes quiet rapidly. At the ordinary temperature solution is rapid, the liquid becomes hot, but not hot enough for the reaction to become tumultuous. As the acid is diluted the attack becomes weaker. With normal acid (83.5 grms. per litre) solution is insignificant at the ordinary temperature (about 0.02 grm. per half hour or per hour).

Except in the case of gas being given off, which makes the action irregular, the latter may be represented by the equation—



The chloric acid in excess is rapidly saturated by the oxide of copper formed, so that the complete reaction is as follows:—



In the solution of the copper by chloric acid we observe that the ratio of the weight of the copper dissolved to the weight of chlorine passing from the state of chlorate to that of chloride, is about 5.36, as required by the reactions (1) and (2).

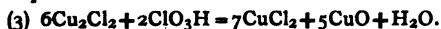
When all the acid is saturated the action of the copper continues on the chlorate thus formed.

In any case no evolution of hydrogen is observed, as has been noticed in the action of chloric acid on zinc or sodium amalgam (O. Dammer, "Handbuch der Anorganische Chemie"). If we electrolyse concentrated chloric acid at about  $60-80^\circ$  with a copper anode, the attack will be naturally very rapid and tumultuous. It is also very rapid, but without the evolution of gas in the case of normal acid.

Under these conditions we also find 5.36 as the ratio of the copper dissolved to the chlorine in the form of chloride. The apparent reaction of the electrolyser thus corresponds to the reactions (1) and (2). The copper dissolved by the current, the same as the copper dissolved by chemical action, passes directly to the state of chloride and oxide; this latter remains in solution, thanks to the excess of chloric acid.

In electrolysis in the cold, principally with a normal solution, we observe that the copper becomes covered with a white deposit which has been identified as cuprous chloride; this deposit is not adherent, and the copper remains perfectly clean. When heated, the metal is brilliant, and there is no deposit of cuprous salt under these circumstances, which is incompatible with the excess of chloric acid.

Cuprous chloride, as I have been able to observe, reduces normal chloric acid fairly rapidly in the cold. If the chloride is in excess, there is a formation of complex basic salts; if the acid is in excess everything is dissolved. When hot the reaction is instantaneous; it is represented by the equation—



With concentrated acid, even in the cold, the action is complicated by the disengagement of oxidised compounds of chlorine. In the action of normal chloric acid on copper without using the electric current, the metal becomes covered with a yellow deposit of the cuprous hydrated oxide, which behaves in the same manner as the chloride, and gives a reaction analogous to (3). The action of chloric acid on copper, with or without electrolysis, such as it is represented by the equations (1) and (2), is thus not

quite complete; it is, in fact, brought about, as we have just seen, by the intermediary of the cuprous compounds.

For these experiments I made use of the same apparatus as for those already published (*Bull. Soc. Chim.*, [3], vol. xxix., p. 156).

The apparatus consists of a porcelain paint-pot 500 c.c. in capacity. This kind of vessel has the drawback of not being transparent, though this is not of much consequence when heating on the water-bath; on the other hand, it has the advantage of being very solid and stable, thanks to the collar which enables us to put it directly into a ring on the water-bath; this is specially convenient with electrolytic operations where we often have to use systems of mechanical agitation connected with complicated apparatus. One of the electrodes is fixed to the pot and the other, generally the anode, to a stirrer worked by a hot-air motor.

The paint-pot is slightly conical, with an available height of 11 or 12 c.m., 7 c.m. diameter at the base, and 8 at the top, so that strips of metal 5 c.m. wide and 15 c.m. long can be used without danger of a short circuit, the available surface being 1 square decimetre. The amount of liquid used for each operation is 450 c.c. When a porous pot is required it should be 14.5 c.m. high and 4.5 c.m. diameter. In such a case we can have 200 c.c. in each compartment with a revolving electrode of 2.5 c.m. by 15 c.m. with an available surface of 50.2 square c.m.

My experiments, generally with a current of 4 ampères, were carried out on chloric acid, chlorate of potassium, and chlorate of copper acidulated with sulphuric acid. The chloric acid was obtained by mixing boiling solutions of chlorate of barium and sulphuric acid at 1 grm. molecule per litre, a slight excess of the former being used so as not to have free sulphuric acid in the final solution, which is about normal, after filtration. For the experiments with acid chlorate of potassium I used a solution containing 50 grms. of  $\text{ClO}_3\text{K}$  and 60 grms. of  $\text{SO}_4\text{H}_2$  per litre.

The chlorate of copper was obtained by mixing two boiling solutions of chlorate of barium and sulphate of copper at 1 grm. molecule per litre, leaving a slight excess of sulphate of copper, and, after filtration and cooling, adding 60 grms. per litre of sulphuric acid.

These different liquids attack the copper in the same manner. Their action under the influence of electrolysis is the same. A platinum cathode was used in every case. The electromotive force, at first very low (0.2 to 0.3 volt), becomes gradually higher with chloric acid and chlorate of copper; with chlorate of potassium it remains practically constant.

In the case of the electrolysis of the acid solution of chlorate of copper, we notice at the commencement a deposit of copper on the cathode, but this soon disappears.

We may remark that in all these experiments the anode is attacked in a very regular manner instead of being perforated, as in the case of the electrolysis of chlorate of potassium.

Thus these different experiments show that copper, in the presence of chloric acid, is dissolved in the cuprous state, but that the salt thus formed changes more or less rapidly into cupric compounds under the influence of chloric acid in excess.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 6.

**The Stolzenberg System of Keeping Notes.**—The Stolzenberg system of filing documents was originally devised as a convenient adjunct to, or substitute for, the complicated system of book-keeping in vogue in many establishments. For some time past we have used the files and cabinets as a convenient means of storing notes of laboratory work and results in a form which renders them readily accessible when wanted; while additions in chronological order, or in that of subject-matter, can be inserted whenever desired. From experience we think that men of science and all engaged in original work will derive benefit from keeping their notes of researches in this systematic and convenient manner.

## RADIO-ACTIVE MINERALS AND SUBSTANCES.

We have received the following communication from the United States Geological Survey:—

The United States Geological Survey is collecting information concerning the occurrence of radio-active minerals and substances in the United States, and would be pleased to have your co-operation. The minerals named on the appended list possess radio-active properties in various forms and degrees. Radio-activity has been noticed also in many substances which are not primarily minerals, such as slags, tailings from concentrators, slimes, chemical wastes, natural mineral waters, deep-well waters, and petroleum, and it is possible that the list of radio-active minerals and substances may be greatly increased. Anyone who has found such minerals or has observed radio-activity in any other substance is urged to give the Survey full details regarding the locality where those occurrences were noted. Any information which the Survey may be able to give will be cheerfully furnished.

The simplest means of detecting radio-activity in a substance is by the use of a photographic plate. The more sensitive the plate the better. The plate should not be removed from the enclosing black paper, and a metal object should be laid upon this black paper in a dark room; upon this should be placed the specimen to be tested. Instead of the metal object a few small nails may be arranged so as to form the initial of the owner and left on the paper-covered plate below the specimen. The specimen should be left in the dark room for from two to fifteen hours, and then developed in the usual manner. If the specimen has radio-active powers, a photograph of the metal object or of the nail-formed initial will be produced on the plate exactly as if it had been exposed to the sun's rays. The test should be made, if possible, with from half a pound to a pound of the material. The electrical method is more reliable, but is much more difficult.

Each specimen submitted should be plainly labelled with the name and post-office address of the sender, the name of the mine or claim from which it came, and the State, county, city, village, mountain, or district in which the deposit is located. If it is desired that the specimens be returned, it must be definitely so stated. On request the Survey will mail postal franks, which will enable the exhibitors to send free of postage a box of specimens weighing not more than four pounds. No specimens will be examined until the finder has first submitted a radio-graph made as described in the preceding paragraph.

Interesting specimens are especially desired for the Survey's exhibit at the Louisiana Purchase Exposition to be held in St. Louis, Mo., from April 30 to December 1 of the present year. This exhibit will be in the United States Government Building, and will be general and varied in character. It will include specimens of every known radio-active substance, whether obtained from minerals and ores, from mineral waters, or from petroleum wells. There will be shown also authentic specimens of radium compounds that have been made by noted investigators. Everything relating to the source, manufacture, and application of radium will be exhibited, including all chemicals obtained from the separation of various radium compounds, and all instruments and devices with which it is proposed to apply radio-activity in medicine, science, and the arts. An interesting feature will be the portraits and the publications of celebrated radium discoverers and investigators, together with photographs of their laboratories and apparatus, and autograph letters from some of them.

Two convenient halls will be set aside for demonstration of the properties of radium. In one will be grouped the specimens of ores and minerals containing radium, and careful note will be made of their effects upon various substances. In the other hall illustrated lectures will be given twice daily on a variety of subjects relating to the history, nature, and possibilities of radium. Its mode of occurrence,

the methods used in separating it from its ores, the concentration of activities from low to high, and the manifold uses to which these remarkable radio-active substances may be put will all be described. Cinematograph Hall will be so arranged that it can be easily darkened, and different highly active specimens of radium compounds will be exhibited in it as affecting the diamond, willemite, kunzite, and other radio-responsive substances.

Investigators of radium are cordially invited to notify the Survey of any material that they would like to exhibit or to put upon record. If they will kindly state the size and character of their material and the space needed for its display, every effort will be made to arrange for its immediate transportation and exhibition.

As the Exposition opened April 30, 1904, it is necessary that prompt action be taken concerning any specimens which are believed worthy of exhibit. All communications regarding the collection and examination of radio-active specimens by the Survey, and concerning its radium exhibit at St. Louis, should be addressed to Mr. George F. Kunz, No. 40, East 25th Street, New York.

CHAS. D. WALCOTT, Director.

## LIST OF MINERALS FOUND TO POSSESS RADIO-ACTIVE PROPERTIES.

## Minerals containing Uranium.

| Name.                         | Percentage of uranium oxide contained. | Radio-activity (Curie's list), uranium unit 1. | No. in Dana's Mineralogy. |
|-------------------------------|----------------------------------------|------------------------------------------------|---------------------------|
| Uranothallite .. ..           | 35—37                                  | —                                              | 307                       |
| Liebigite .. ..               | 36—38                                  | —                                              | 308                       |
| Voglite .. ..                 | 37                                     | —                                              | 309                       |
| Thorite .. ..                 | 1—10                                   | 0.7—2                                          | 395                       |
| Var. Uranothorite .. ..       | —                                      | —                                              | —                         |
| Rowlandite .. ..              | 0.04                                   | —                                              | —                         |
| Uranophane .. ..              | 53—67                                  | —                                              | 503                       |
| Hatchettolite .. ..           | 15—16                                  | —                                              | 521                       |
| Fergusonite .. ..             | 1—3                                    | 0.1—0.4                                        | 523                       |
| Var. Tyrite .. ..             | 5—6                                    | —                                              | —                         |
| „ Bragite .. ..               | 8—9                                    | —                                              | —                         |
| Spyllite .. ..                | 3—4                                    | 0.1—0.3                                        | 524                       |
| Yttrotantalite .. ..          | 1—2                                    | —                                              | 528                       |
| Samaraskite .. ..             | —                                      | 1.1                                            | —                         |
| Var. — .. ..                  | 9—16                                   | —                                              | 529                       |
| Annerödite .. ..              | 16—17                                  | —                                              | 530                       |
| Hielmite .. ..                | 2—5                                    | —                                              | 531                       |
| Euxenite .. ..                | 5—12                                   | —                                              | 534                       |
| Polycrase .. ..               | 5—20                                   | —                                              | 535                       |
| Arhenite .. ..                | 23—24                                  | —                                              | 535½                      |
| Xenotime .. ..                | 0—4                                    | 0.03                                           | 536                       |
| Torbernite .. ..              | 57—62                                  | —                                              | 659                       |
| Zeunerite .. ..               | 55—56                                  | —                                              | 660                       |
| Autunite .. ..                | 55—62                                  | 2.7                                            | 661                       |
| Uranospinite .. ..            | 59—60                                  | —                                              | 662                       |
| Uranocircite .. ..            | 56—57                                  | —                                              | 663                       |
| Phosphuranylite .. ..         | 71—76                                  | —                                              | 664                       |
| Trögerite .. ..               | 63—64                                  | —                                              | 665                       |
| Walpurgite .. ..              | 20—21                                  | —                                              | 666                       |
| Carnotite .. ..               | 62—65                                  | —                                              | —                         |
| Uraninite (pitchblende) .. .. | 75—85                                  | 1.6—8.3                                        | 711                       |
| Var. Bröggerite .. ..         | Vary in                                | —                                              | —                         |
| „ Cleveite .. ..              | ratio of                               | —                                              | —                         |
| „ Nivenite .. ..              | uranium                                | 1.4                                            | —                         |
| „ Urannibolite .. ..          | oxide.                                 | —                                              | —                         |
| Gummite .. ..                 | 61—75                                  | —                                              | 712                       |
| Var. Thorogummite .. ..       | —                                      | —                                              | —                         |
| „ Yttrogummite .. ..          | —                                      | —                                              | —                         |
| Mackintoshite .. ..           | 21—22                                  | —                                              | —                         |
| Uranosphærite .. ..           | 50—51                                  | —                                              | 713                       |
| Johannite .. ..               | 67—68                                  | —                                              | 806                       |
| Uranopilite .. ..             | 77—78                                  | —                                              | 807                       |

Minerals containing Thorium.

(It has been pretty well established that thorium is not radioactive unless the mineral in which it is found contains uranium also).

| Name.                     | Percentage of thorium oxide contained. | Radio-activity (Curie's list), uranium unit 1. |
|---------------------------|----------------------------------------|------------------------------------------------|
| Tysonite .. .. .          | —                                      | —                                              |
| Fluocerite .. .. .        | —                                      | —                                              |
| Yttrocerite .. .. .       | —                                      | —                                              |
| Cassiterite .. .. .       | —                                      | —                                              |
| Var. Ainalite .. .. .     | —                                      | —                                              |
| Parisite .. .. .          | —                                      | —                                              |
| Bastnaesite .. .. .       | —                                      | —                                              |
| Lanthanite .. .. .        | —                                      | —                                              |
| Tengerite .. .. .         | —                                      | —                                              |
| Lavenite .. .. .          | —                                      | —                                              |
| Wohlerite .. .. .         | —                                      | —                                              |
| Eudialyte .. .. .         | —                                      | —                                              |
| Var. Eucolite .. .. .     | —                                      | —                                              |
| Cappelenite .. .. .       | —                                      | —                                              |
| Melanocerite .. .. .      | 1—2                                    | —                                              |
| Caryocerite .. .. .       | 13—14                                  | —                                              |
| Tritomite .. .. .         | 8—9                                    | —                                              |
| Zircon .. .. .            | 0—2                                    | —                                              |
| Var. Tachyaphalite ..     | Small amount                           | —                                              |
| „ Cyrtolite .. .. .       | trace                                  | —                                              |
| Thorite .. .. .           | 48—72                                  | 0·7—2                                          |
| Var. Auerlite .. .. .     | 70                                     | —                                              |
| „ Calciorthorite .. ..    | 59—60                                  | —                                              |
| „ Eucrasite .. .. .       | 35—36                                  | —                                              |
| „ Freyalite .. .. .       | 28—29                                  | —                                              |
| „ Orangite .. .. .        | 71—72                                  | 2                                              |
| „ Uranothorite .. ..      | 52                                     | —                                              |
| Gadolinite .. .. .        | 0—1                                    | —                                              |
| Var. Metagadolinite ..    | —                                      | —                                              |
| Yttrialite .. .. .        | 12                                     | —                                              |
| Thalenite .. .. .         | —                                      | —                                              |
| Allanite .. .. .          | —                                      | —                                              |
| Var. Bodenite .. .. .     | —                                      | —                                              |
| „ Muromonite .. .. .      | —                                      | —                                              |
| „ Orthite .. .. .         | —                                      | —                                              |
| „ Uralorthite .. .. .     | —                                      | —                                              |
| Cerite .. .. .            | —                                      | —                                              |
| Cenosite .. .. .          | —                                      | —                                              |
| Keilhauite .. .. .        | —                                      | —                                              |
| Tscheffkinite .. .. .     | 0—21                                   | —                                              |
| Johnstrupite .. .. .      | trace                                  | —                                              |
| Mosandrite .. .. .        | trace                                  | —                                              |
| Rinkite .. .. .           | —                                      | —                                              |
| Dysanalite .. .. .        | —                                      | —                                              |
| Pyrochlor .. .. .         | 8                                      | —                                              |
| Hatchettolite .. .. .     | —                                      | —                                              |
| Microlite .. .. .         | —                                      | —                                              |
| Fergusonite .. .. .       | —                                      | 0·1—0·4                                        |
| Var. Kochelite .. .. .    | 1—2                                    | —                                              |
| Sipylite .. .. .          | —                                      | 0·1—0·3                                        |
| Columbite (tantallite) .. | —                                      | 0·02                                           |
| Tapiolite .. .. .         | —                                      | —                                              |
| Skogbolite .. .. .        | —                                      | —                                              |
| Stibiotantalite .. .. .   | —                                      | —                                              |
| Mossite .. .. .           | —                                      | —                                              |
| Yttrotantalite .. .. .    | —                                      | —                                              |
| Samarските .. .. .        | —                                      | 1·1                                            |
| Var. From Colorado ..     | 3—4                                    | —                                              |
| Annerödite .. .. .        | 2—3                                    | —                                              |
| Hielmite .. .. .          | —                                      | —                                              |
| Æschynite .. .. .         | 15—17                                  | 0·7                                            |
| Polymignite .. .. .       | 3—4                                    | —                                              |
| Zirkelite .. .. .         | 7—8                                    | —                                              |
| Euxenite .. .. .          | 0—6                                    | —                                              |
| Polycrase .. .. .         | 3—4                                    | —                                              |
| Arrhenite .. .. .         | —                                      | —                                              |
| Rogersite .. .. .         | —                                      | —                                              |
| Xenotime .. .. .          | 0—3                                    | 0·03                                           |
| Monazite .. .. .          | 0—18                                   | 0·5                                            |

Minerals containing Thorium (continued).

| Name.                      | Percentage of thorium oxide contained. | Radio-activity (Curie's list), uranium unit 1. |
|----------------------------|----------------------------------------|------------------------------------------------|
| Rhabdophanite .. .. .      | —                                      | —                                              |
| Churchite .. .. .          | —                                      | —                                              |
| Uraninite (Pitchblende) .. | 0—10                                   | 1·6—8·3                                        |
| Var. Bröggerite .. .. .    | 5—6                                    | —                                              |
| „ Cleveite .. .. .         | 3—5                                    | 1·4                                            |
| „ Nivenite .. .. .         | 6—8                                    | —                                              |
| „ Urannibate .. .. .       | —                                      | —                                              |
| Gummite .. .. .            | (0—42)                                 | —                                              |
| Var. Thorogummite ..       | 41—42                                  | —                                              |
| „ Yttrogummite .. .. .     | 0—?                                    | —                                              |
| Mackintoshite .. .. .      | 45—46                                  | —                                              |

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 18th, 1904.

Prof. W. A. TILDEN, D.Sc., F.R.S., President, in the Chair.

BEFORE entering on the ordinary business of the meeting, the PRESIDENT said he thought it would be consonant with the wishes of the Fellows if he gave expression to the deep feeling of regret with which they had all received the news of the death of one of the most eminent of their past Presidents, Professor Williamson. A man of philosophic mind and great intellectual activity, he had been led in the later years of his life to abandon research in connection with chemistry in favour of other pursuits. Hence it was difficult for chemists of the present generation to realise how great had been the influence exercised by his experimental researches and his writings on the progress of theoretical chemistry forty years ago. Williamson served the Society during many years on the Council, as vice-President, and for two separate periods as President. The Society had, of course, been represented at the funeral, at which many past and present officers of the Society—including himself and the Treasurer—had assembled, and a resolution of condolence with the family had that day been passed at the meeting of the Council.

Messrs. N. J. Bluman, J. C. Evans, G. Pinchbeck, and M. W. Stevens were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Frank Harold Lowe, B.Sc., 59A, Fulham Park Gardens, S.W.; Henry Stanley Shelton, 4, Larden Road, Acton Vale, W.; Herbert Jenkins, 52, Criffell Avenue, Streatham Hill, S.W.; Rudolf Lessing, Ph.D., 31, Brunswick Square, W.C.; Robert Rodger, 54, Rostrevor Road, S.W.; William Edward Oakden, 2, Gledhow Terrace, South Kensington, S.W.; Charles John Sawyer, 6, Cleveland Road, Brighton.

A ballot for the election of Honorary and Foreign members was held, and the following were subsequently declared duly elected:—Prof. Antoine Henri Becquerel, Prof. Cornelis Adrian Lobry de Bruyn, Prof. Frank Wigglesworth Clarke, Madame Marie Curie, Prof. Carl Theodor Liebermann, Prof. Edward Williams Morley.

In reply to Prof. Divers, the PRESIDENT stated that Counsel's opinion had been taken on the question whether women were eligible for election as ordinary Fellows of the Society under the present Charter, and that the opinion was adverse.

\*87. "The Action of Nitrosyl Chloride on Pinene." By WILLIAM AUGUSTUS TILDEN.

Pinene nitroschloride having been shown by von Baeyer to have the double formula  $(C_{10}H_{16}NOCl)_2$ , it occurred to the author that the unsatisfactory yield of this compound by the usual processes might be improved by using a mixture

of equal quantities of *d*- and *l*-pinenes which is optically inactive. Whereas previously the yield of nitrosochloride from ordinary *d*-pinene was about 32 per cent of the pinene, and the product from the more highly rotatory pinene obtained from French turpentine was still less, the employment of a mixture prepared so as to be optically inactive gives 55 per cent.

The melting point of pure pinene nitrosochloride is not  $103^{\circ}$ , as originally given, but  $115^{\circ}$  (*circa*). Attempts to resolve pinylamine into two optically active bases proved unsuccessful.

For the regeneration of pinene from the nitrosochloride, methylaniline is recommended in preference to aniline, as there is no violent action and the yield of pinene is greater.

Finally, it is shown that when the nitrosochloride is converted into nitrosocyanide (Tilden and Burrows, *Proc.*, 1902, xviii., 161) or into the piperidynitrolamine, the compound becomes monomolecular at the same time that the nitroso-group assumes the *isonitroso*- or *oxine* structure.

#### DISCUSSION.

Professor MELDOLA suggested that the base produced by the action of dimethylaniline on pinene nitrosochloride might be Bindecheller's green resulting from the secondary interaction between nitrosodimethylaniline and the excess of dimethylaniline present under the conditions of the experiment (*Ber.*, 1883, xvi., 864).

\*88. "The Electrolytic Estimation of Minute Quantities of Arsenic." By HENRY JULIUS SALMON SAND and JOHN EDWARD HACKFORD.

A high supertension of the cathode is requisite for the reduction of arsenic acid to metallic arsenic, the reaction being most readily effected in the presence of metals having this property, such as lead or zinc and probably also mercury. Platinum, having a low supertension, is quite inefficient.

The production of arsenic trihydride from arsenites is accomplished more readily by platinum cathodes than by those of copper, which have a much higher supertension. In this case, mercury, with an extremely high supertension, is quite unsuitable. A certain amount of supertension, however, appears to be necessary, for platinised platinum with no supertension is inefficient.

The authors recommend the use of lead electrodes for the estimation of minute quantities of arsenic, as their application causes a simplification of previous methods. Errors which may arise in the electrolytic methods owing to the presence of foreign metals can be rectified by the addition of lead acetate or zinc sulphate to the electrolyte except when the foreign metal is mercury. When lead and zinc cathodes are used, the smallest amount of arsenic which can be detected in alkaline solutions of arsenates and arsenites is about thirty times as great as in acid solution, but platinum cathodes are quite unsuitable.

#### DISCUSSION.

Dr. F. M. PERKIN remarked that although the electrolytic apparatus gave good results even with very minute quantities of arsenic, it was, however, a great advantage to have an apparatus in which lead was employed for the cathode instead of the very expensive platinum. The fact that a mercury cathode did not give good results, although there was considerable over-voltage, was due to the formation of an arsenic amalgam, which then escaped the reducing action of the hydrogen. An aluminium cathode is suitable for dilute sulphuric acid solutions, because in this case there is considerable over-voltage and the metal is very slightly attacked, and is, moreover, free from arsenic.

Had nickel electrodes been tried in an alkaline solution?

The only drawback to the electrolytic method for detecting arsenic is the trouble of preparing the mirrors, especially when special standards are made for each substance tested.

Dr. STEVENSON asked what was the authors' evidence

that the lead of commerce can be obtained free from arsenic.

Mr. SAND, in reply, said the electrolytic experiments in alkaline solutions had not been found satisfactory, and had, moreover, not been tried in the case of nickel.

The preparation of mirrors for each kind of material examined was not absolutely necessary, but, nevertheless, this precaution rendered the comparisons more certain.

Several grms. of commercial lead had been examined for arsenic with a negative result. The lead most frequently used for cathodes was purest assay lead, whereas pure commercial lead was employed for anodes. New electrodes often contain a small amount of arsenic on the surface, but in all cases they were tested by blank experiments.

\*89. "The Action of Sodium Methoxide and its Homologues on Benzophenone Chloride and Benzylidene Chloride." Part II. By JOHN EDWIN MACKENZIE and ALFRED FRANCIS JOSEPH.

The authors have found that benzhydrol is formed in the preparation of diisopropoxy-, diisobutoxy-, and diisoamyloxy-diphenylmethanes by the action of the respective sodium alkylloxides on benzophenone chloride (compare *Trans.*, 1901, lxxix., 1204). It is also shown that dibenzoyldiphenylmethane, like its homologues, readily splits up into benzophenone and dibenzyl oxide.

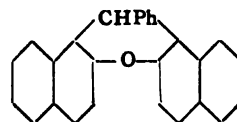
The anhydride of phenyldi- $\beta$ -hydroxynaphthylmethane has been obtained by the action of benzylidene chloride on  $\beta$ -naphthol, and found to agree in properties with the substance prepared by Claisen (*Annalen*, 1887, cccxxvii., 261) from benzaldehyde and  $\beta$ -naphthol. This product was also obtained when the reaction was carried out in xylene solution and when sodium naphthoxide was used instead of naphthol. Claisen's dihydroxy-compound could not be isolated.

Nitration experiments on the anhydride led to the formation of substances containing from two to six nitro-groups.

When treated with fuming sulphuric acid, the anhydride yielded a  $\beta$ -naphtholdisulphonic acid, but concentrated sulphuric acid gave rise to a product dissolving in water to a bright red solution, the colour of which was destroyed by alkalis and restored by acids.

#### DISCUSSION.

Professor MELDOLA said that in his opinion the constitution of Claisen's compound might be more correctly represented by the following formula:—



as it was more in harmony with the general properties of  $\beta$ -naphthol that the  $\alpha$ -ortho-position, and not the second  $\beta$ -position, should be first attacked.

Dr. HEWITT stated that the hydroxylic compound,  $C_6H_5CH(C_{10}H_6OH)_2$ , is easily produced by condensing benzaldehyde and  $\beta$ -naphthol in cold acetic acid solution by the aid of hydrochloric acid, and from this the anhydrous substance can be obtained in good yield by heating with acetic acid in sealed tubes.

The colouration observed on dissolving the anhydride in strong sulphuric acid is probably due to oxidation and formation of a carboxonium salt similar to those already studied by Werner, Fosse, and himself.

Dr. MACKENZIE replied that at present he had no decided views in favour of the 2:3-structure of the anhydride molecule as against the 1:2-configuration; he had not yet obtained any substitution products in support of either of these constitutions.

\*90. "The Bromination of Phenolic Compounds." By JOHN THEODORE HEWITT, JAMES KENNER, and HARRY SILK.

When one molecular proportion of bromine acts on phenol, the character and proportions of the products obtained vary with the conditions under which the reaction is carried out. In aqueous solution, the phenol is apparently not all brominated, some of the bromine being used in forming higher substitution products. Absence of water and presence of a strong mineral acid favours the formation of *p*-bromophenol; the quantity of the para-compound diminishes, however, if sodium acetate is added to a glacial acetic acid solution of phenol before bromination, but is slightly increased by the addition of concentrated sulphuric acid.

If more than one molecular proportion of bromine is added to a solution of phenol in concentrated sulphuric and glacial acetic acids, the second molecule of bromine is utilised very slowly, the sulphuric acid hindering substitution in the ortho-position, but by brominating phenol in excess of 73 per cent sulphuric acid, the halogen rapidly enters into reaction, and a nearly quantitative yield of 2:4-dibromophenol is obtained, apparently without any admixture of tribromophenol.

5-Bromosalicylic acid can be conveniently prepared by brominating salicylic acid in a mixture of sulphuric and acetic acids.

4-Bromo-*o*-naphthol cannot, however, be obtained from *o*-naphthol under similar conditions, dibromination taking place.

#### DISCUSSION.

Professor MELDOLA pointed out that in the case of the naphthols it was well known that the first action of bromine is to produce additive products from which hydrogen bromide is expelled by heat. With reference to the influence of temperature as determining the formation of *o*-bromophenol instead of the para-compound, he called attention to Merck's German patent (No. 76,597, of 1893), in which it is claimed that by chlorinating or brominating phenol at 150—180° the corresponding halogenated ortho-derivative is produced.

91. "The Decomposition of the Alkylureas." (Preliminary Note.) By CHARLES EDWARD FAWSITT.

The author has already shown (*Zeit. physikal. Chem.*, 1902, xli., 601) that the decomposition of urea by acids is not a case of ordinary hydrolysis, but is the result of a secondary decomposition, following an isomeric transformation into ammonium cyanate. An investigation of the velocity of decomposition of the alkylureas with acids by similar methods shows an exact parallel with the case of urea. The hydrolysis is indirect, and is effected as a secondary reaction of the acid with the alkylammonium cyanate. Methylurea is decomposed more slowly than urea, but dimethylurea much more rapidly.

\*92. "The Formation of Periodides in Nitrobenzene Solution. Part II. Periodides of the Alkali and Alkaline Earth Metals." By HARRY MEDFORTH DAWSON and ETHEL ELIZABETH GOODSON.

Previous experiments relating to the nature of the periodides which are formed in nitrobenzene solutions containing iodine and potassium iodide (Dawson and Gawler, *Trans.*, 1902, lxxxi., 524) have been extended to the iodides of the other alkali metals, and to those of the alkaline earth metals, and ammonium and substituted ammoniums. In general, these iodides have properties similar to those of the potassium derivative, and the experimental data indicate that enneaiodides of the type  $M'I_9$  or  $M''I_8$ , which are the essential components of such solutions saturated with iodine, probably represent the highest limiting type of periodide, for the solvent used in these experiments appears to be specially suited to the formation of such periodides, and saturation of the solution with iodine favours the formation of the most complex product.

In the case of certain substituted ammonium radicles, enneaiodides have been already isolated, and the analogous behaviour of the alkali and alkaline earth metals indicates

the possibility of preparing enneaiodides of these metals under favourable conditions.

Periodides are also formed by the bromides of the alkali metals in nitrobenzene solution and to a small extent by the chlorides.

Compounds of lithium iodide with nitrobenzene and *o*-nitrotoluene, and of sodium penta-iodide with nitrobenzene have been isolated. -

93. "The Action of Ozone on Ethane." (Preliminary Note.) By WILLIAM ARTHUR BONE and JULIEN DRUGMAN.

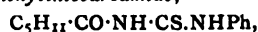
The authors have obtained ethyl alcohol by the interaction of ethane and ozone at 100°. Two experiments have been carried out as follows:—Ethane and ozonised air ( $O_3$  = about 2 per cent) were separately led into the top of a vertical, wide glass tube, about 18 inches long, packed with glass beads and heated by a steam jacket. The proportions of the gases were so regulated that the ethane was always present in large excess, under which conditions the ozone entirely disappeared as the mixture slowly descended the tube. The gases were then drawn through a series of cooled glass worms containing water for the absorption of soluble intermediate products. Each experiment extended over three or four days, during which about 5 litres of ethane and 13 to 15 litres of the ozonised air passed through the apparatus. Subsequent examination of the liquid from the coolers showed that it contained ethyl alcohol, acetaldehyde, and traces of formaldehyde. The presence of ethyl alcohol was proved by first oxidising the aldehydes by means of an excess of an ammoniacal silver solution at the ordinary temperature (one hour), then acidifying the liquid with dilute sulphuric acid and submitting it to distillation in steam. The distillate in each case at once gave the iodoform reaction; the precipitate was composed of microscopic crystals, which slowly formed the characteristic star-like aggregates. As a precaution, the absence of acetaldehyde from the distillate was proved by the negative result obtained with Schiff's reagent. An examination of the gaseous products showed that they did not contain acetylene, ethylene, or free hydrogen.

There can be but little doubt, therefore, that ethyl alcohol is the primary product in the slow combustion of ethane. At temperatures where ethane begins to react with oxygen with appreciable velocity, the alcohol is oxidised so many times faster that it is practically impossible actually to detect its formation (compare Bone and Stockings, *Proc.*, xx., p. 106). The authors propose to continue the experiments, and to extend them to ethylene, acetylene, and other typical hydrocarbons.

94. "Caproylthiocarbimide." By AUGUSTUS EDWARD DIXON.

Caproyl chloride, dissolved in benzene, interacts spontaneously with finely-powdered ammonium thiocyanate, yielding caproylthiocarbimide,  $C_5H_{11}CO \cdot NCS$ ; b. p. 108°/23 mm.; sp. gr. = 1.0165 at 18°/15°. This liquid is slowly hydrolysed by boiling water into caproic and thiocyanic acids; when heated with an alkaline lead salt, it yields metallic sulphide, together with thiocyanate, and is readily desulphurised by ammoniacal silver nitrate. If brought into contact with primary and secondary amines, direct union occurs, with formation of the corresponding thiocarbamides.

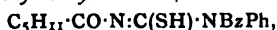
ab-Caproylphenylthiocarbamide, —



separates from alcohol in felted masses of needles, melting at 77—78° without decomposition; when dissolved in hot dilute caustic alkali, the caproyl group is removed, leaving phenylthiourea.

ab-Caproyl-*o*-tolylthiocarbamide melts at 97—98° and generally resembles the phenyl compound. When treated in hot alcoholic solution with the calculated quantity of silver nitrate, it yields the corresponding ab-caproyl-*o*-tolylurea (m. p. 99—100°). ab-Caproyl-*p*-tolylthiocarb.

amide (m. p. 90—91°), when treated with silver nitrate, yields caproyl-p-tolylurea, melting at 131—132° (corr.).  
n-Caproylphenylbenzylthiourea,—



was obtained from benzylaniline and the thiocarbimide; it separated from alcohol in vitreous prisms melting at 77—78°, and was not affected by boiling with neutral or ammoniacal silver nitrate; when boiled with caustic alkali, the caproyl group is eliminated, leaving a residue which can be desulphurised by an alkaline solution of lead.

Since, by interaction with water, the parent oil gives thiocyanic acid instead of carbon oxysulphide, its behaviour in these circumstances must be accounted that of a thiocyanate; on the other hand, the power to combine with nitrogenous bases, thereby yielding substituted thiocarbamides, places it among the isothiocyanates. This dual capacity is in accordance with the view developed in earlier papers by the author (*Trans.*, 1901, lxxix., 542; 1904, p. 350), that a variety of tautomerism subsists amongst the thiocyanates of electronegative radicles, the characteristic sulphuretted group of which may act either as  $\cdot\text{SCN}$  or  $\cdot\text{NCS}$ . Moreover, in certain cases, both forms appear to be simultaneously active (Doran, *Proc.*, 1904, xx., 20), the preponderance of one or other depending almost entirely on the temperature at which interaction is caused to take place.

## NOTICES OF BOOKS

*A Text-book of Ceramic Calculations.* With Examples. By W. JACKSON, A.R.C.S. London, New York, and Bombay: Longmans, Green, and Co. 1904. Pp. 67.

THIS book has been written primarily for the use of the classes in pottery and porcelain manufacture, held under the direction of the Staffordshire Education Committee. It contains a good deal of practical as well as theoretical instruction on compounding mixtures of known composition, the application of such methods to the complete synthesis of mixtures, &c., and the general subject of pottery making. The rational analysis of clay, and the application of this method to the synthesis of bodies are also dealt with in two chapters. There is a complete list of elements, compounds, and minerals of importance in pottery manufacture, with their formulæ, composition, &c., but there is no index.

*Report on the Agricultural Work in the Botanic Gardens and the Government Laboratory at British Guiana for the Year 1902-1903.* Georgetown, Demerara: C. K. Jardine, Printer to the Government.

THE bulk of this report consists of tables of results obtained from the experiments on the effect of various manures on sugar-cane growing. The general deductions as to the action of these manures are that nitrogen in the form of sulphate of ammonia, nitrate of soda, &c., is without doubt the manurial constituent, the supply of which mainly governs the yield of the plant; on the whole the former is the preferable salt to employ, and dressings of 2 to 3 cwt. of sulphate of ammonia per acre appear to be the most certainly profitable applications of nitrogen, though in favourable seasons the use of still higher proportions may prove successful; we may remark, however, that it is not until the season is over that it can be known whether it has been a favourable one or not, so how is the planter to know when he is to apply the larger amount of sulphate of ammonia? The application of superphosphate of lime in addition to manurings of nitrogen and potash is found to be advantageous with plant-canes only, ratoons should be manured with nitrogen only. The use of lime has resulted in largely increased yields, but whether or not its use is

profitable depends on the price of sugar; it is, however, confirmed that neither the addition of phosphoric acid, potash, or lime to the manures favourably affects the sugar contents of the juice.

*Dictionary of Photographic Chemistry.* ("Dictionnaire de Chemie Photographique"). For the Use of Amateurs and Professionals. By G. and AD. BRAUN fils. In Eight Monthly Parts. Paris: Gauthier-Villars. 1904. Pp. 500. Parts I., II., and III.

THIS dictionary comprises the nomenclature and description of all substances connected with photographic science. After a short general account of each preparation, the authors give its various chemical properties, and lay special stress on any of its applications to photography. All the photographic reactions as well as the most important manipulations are fully described, preference being given to practical operations rather than to theoretical discussions. This dictionary will be of great use to those engaged in photography, either as professionals or as amateurs, who really wish to understand what they are doing, as opposed to merely "pushing the button."

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 18, May 2, 1904.

**Electrolytic Solution of Platinum. New Method for Preparing Platinocyanides.**—André Brochet.—Platinum behaves in the same way as iron and cobalt towards an alternating current, and dissolves very easily in cyanides. This fact is especially interesting, because this metal offers so much resistance to chemical agents. The explanation of this solution is particularly instructive from a theoretical point of view, and the author is further investigating the matter.

**Origin of the Blondlot Rays emitted during Chemical Reactions.**—Albert Colson.—Chemical actions which emit Blondlot rays are always accompanied by physical actions (contraction, cooling, &c.) which act in the same sense. Also as certain brisk reactions—such as precipitation of salts, oxides, &c.,—emit no  $\text{N}$  or  $\text{N}_2$  radiation, apparently there exists no definite relationship between the intensity of the chemical reactions and the emission of these rays. It is just this want of proportion which renders the Blondlot rays of use to the chemist to disclose secondary or delicate reactions, often masked by the brisker reaction.

**Reduction of Silicon by Hydrogen.**—A. Dufour.—Silicon is reduced at a high temperature by hydrogen, producing siliciuretted hydrogen and water. The reverse reaction is possible. This reduction explains the phenomenon of the apparent devitrification of silicon bulbs when they are blown by tubes. This reaction also gives a satisfactory explanation of Boussingault's and Schützenberger's experiments on silicuration of platinum by silicon in an atmosphere of hydrogen.

**Zinc-aluminium Alloys.**—Hector Pécheux.—The author tries to unite zinc and aluminium in all proportions, and he succeeds in isolating nine fairly definite alloys, corresponding to the following formulæ:— $\text{Zn}_3\text{Al}$ ,  $\text{Zn}_2\text{Al}$ ,  $\text{ZnAl}$ ,  $\text{ZnAl}_2$ ,  $\text{ZnAl}_3$ ,  $\text{ZnAl}_4$ ,  $\text{ZnAl}_6$ ,  $\text{ZnAl}_{10}$ , and  $\text{ZnAl}_{12}$ . He melts these in cylindrical ingots of 1 c.m. diameter, and in spheres 2.70 m.m. diameter. The fusion of these alloys presents an interesting peculiarity, which explains the great heat of fusion of zinc, 28.13°. When solid zinc



is thrown into melted aluminium (650°), the zinc being at the ordinary temperature, the latter melts slightly in contact with the aluminium, then all the aluminium solidifies. The mixture has then to be re-heated to obtain the fusion of the two metals, which afterwards mix intimately.

**Action of Diazobenzene Chloride on Diphenylamine.**—Léo Vignon and A. Simonet.—The body obtained by the action of diazobenzene chloride on diphenylamine has the constitution of phenyldiazoamidobenzene,  $C_6H_5.N=N.N(C_6H_5)_2$ . The method employed for its preparation can apparently be used for the preparation of a series of new diazoamide bodies, derivatives of diphenylamine and analogous bases.

**Allyl- and Propenylalcoylketones.**—E. E. Blaise.—The author makes a series of researches to absolutely define the constitution of the non-saturated isomeric ketones, some being propenyl and the others allyl.

**Application of Grignard's Reaction to the Halogen Ethers of the Tertiary Alcohols.**—L. Bouveault.—The author succeeds in applying Grignard's reaction to the tertiary chlorides of butyl and amyl, but he establishes the fact that the organo-magnesium derivatives thus obtained only partially obey Grignard's rules.

**Symmetric Bichlormethyl Oxide.**—Marcel Descudé.—Symmetric bichlormethyl oxide can be obtained by the action of chlorine either on methyl oxide or on monochlormethyl oxide. The author showed in a previous research that it is produced as a secondary product by the general action of acid chlorides on polyoxymethylene in presence of zinc chloride. He now further discovers that it can be obtained as the principal product of the reaction of phosphorus trichloride and  $(CH_2O)_n$ .

## MISCELLANEOUS.

**Technological and Scientific Dictionary.**—We have received from the publishers, Messrs. George Newnes, Limited, a copy of Part II. of the new "Technological and Scientific Dictionary" they are now bringing out. This number comprises those subjects between BOW (Bow Compasses) and COP (Copper Glance). The Dictionary contains in addition to many special articles, definitions of the terms used in all the principal scientific and technological subjects, and when complete should form a useful addition to the reference library.

**The Carbonatopentamine Cobaltic Salts.**—A. Werner and N. Goslings.—These salts, which answer to the general formula  $CO_3Co(NH_3)_5X$ , are very soluble and of a brown to violet-red colour; they have only a single one of the carbonate reactions; that is, they give off carbonic acid with acids. *The Nitrate*,  $CO_3Co(NH_3)_5NO_3 + H_2O$ .—We mix a solution of 100 grms.  $(NO_3)_2Co$ , in 50 c.c. of water, with a solution of 150 grms. of carbonate of ammonium in 150 c.c. of water and 250 c.c. of ammonia at 20.5°; expose to the air for twelve hours at the ordinary temperature. Sixty-eight grms. of pointed red crystalline tufts and plates are then deposited; we can re-crystallise these in warm water as bright brownish-red plates. This salt can be obtained in a more pure form by the reaction of  $NO_3Ag$  on the corresponding iodide; after the separation of the AgI, we add alcohol. Red plates are formed which are washed with alcohol and ether, and which are re-crystallised in water on the water-bath. With dilute acids  $CO_2$  is given off. Iodomercurate of potassium gives a precipitate; the ferro- and ferricyanides give a brown precipitate, and after some time  $PtCl_6Na_2$  gives a yellow precipitate, and  $S_2O_6Na_2$  gives a crystalline red one. *The Bromide*,  $CO_3Co(NH_3)_5Br + H_2O$ .—The nitrate is dissolved in a very small quantity of water, and solid KBr added; on the addition of alcohol a brownish-red crystalline precipitate is formed which, when taken up with tepid water, gives large, red, quadratic crystals, very soluble in water, decom-

posing above 80°. *The Iodide*,  $CO_3Co(NH_3)_5I + H_2O$ .—This salt is prepared in the same manner as the bromide; it is a deep red crystalline powder, and after re-crystallisation in water, it forms very soluble red tables.—*Berichte*, vol. xxxvi., p. 2378.

**Compounds of Nitrate of Silver with Ammonia.**—V. Kourilof.—The author only gives the results of his researches, which are as follows:—1. Reyckler's mono-ammoniacal compound,  $AgNO_3 \cdot NH_3$ , cannot be formed from aqueous or alcoholic solutions of ammonia and nitrate of silver, except when we take very accurately determined proportions of the two constituents. 2. The influence on the composition of Reyckler's compound of small variations in the concentration of the nitrate of silver and the ammonia, and the impossibility of crystallising it in alcohol, show that it is a crystalline mixture of  $AgNO_3 \cdot 2NH_3$  and of  $AgNO_3$ . 3. The separation of this crystalline mixture from its alcoholic solutions by the addition of ether, results from the slight solubility of  $AgNO_3 \cdot 2NH_3$  and of  $AgNO_3$ . 4. We know that the solubility of  $AgNO_3$  in alcohol diminishes with the proportion of water present; the saturated solution of  $AgNO_3$  in anhydrous alcohol (density = 0.7993 at 15° C.) contains at 22.9° 0.1754, and at 23.3° 0.1804 molecules per litre. 5. The addition of 2 molecules of ammonia to the saturated solution of nitrate of silver makes the solubility four to six times less (at 22.9° the solubility of  $AgNO_3 \cdot 2NH_3$  is 0.0383 molecule). 6. The solubility of  $AgNO_3 \cdot 2NH_3$  in alcohol increases slowly with the temperature; between 19° and 50° it is represented by an almost straight line (at 19°, 0.0353 molecule; at 30°, 0.0495 molecule). 7. The isotherms of the variation of solubility of  $AgNO_3 \cdot 2NH_3$  in alcohol, for variable quantities of  $NH_3$ , has a characteristic peculiarity; the solubility of  $AgNO_3 \cdot 2NH_3$  increases at first with the proportion of  $NH_3$ , reaches a maximum, then diminishes. 8. Between -14° and +40°, aqueous or alcoholic solutions of nitrate of silver saturated with ammonia, do not separate any other ammoniacal compound than  $AgNO_3 \cdot 2NH_3$ .—*Journ. Soc. Phys. Chim. R.*, vol. xxxiv., p. 843.

## MEETINGS FOR THE WEEK.

- MONDAY, 6th.—Royal Institution, 5. General Monthly Meeting.  
Society of Chemical Industry, 8. "Loss of Nitre in the Chamber Process," by J. K. H. Inghis. "Acetone—its Manufacture and Purification," by A. Marshall. "New Method for the Estimation of Tannin," by Dr. J. Gordon Parker and E. E. M. Payne.  
THURSDAY, 9th.—Faraday Society, 8. "The Hard and Soft State in Metals," by G. T. Bellby. "The Electric Furnace—its Origin, Transformations, and Applications," by Adolphe Minet.

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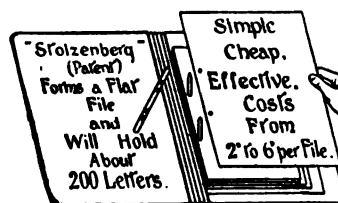
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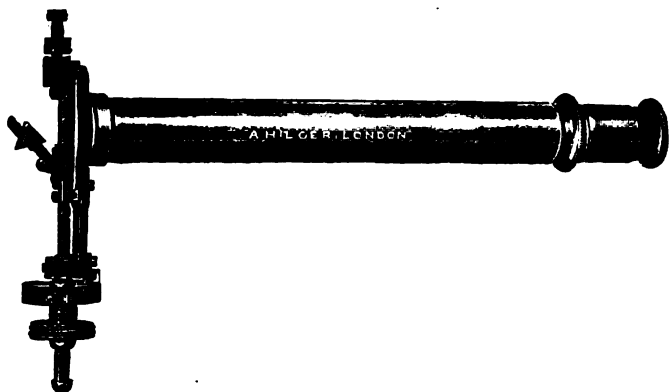
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### CONTENTS.

| ARTICLES:—                                                                                                                                      | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------------|------|
| Preparation of Samaria and Atomic Weight of Samarium, by G. Urbain and H. Lacombe .....                                                         | 277  |
| A New Method for the Fractionation of the Cerite Salts, by H. Lacombe .....                                                                     | 277  |
| Influence of the Physical Nature of the Anode on the Constitution of Electrolytic Peroxide of Lead—Application to Analysis, by A. Hollard ..... | 278  |
| The Formation of Basic Salts of Copper under the Influence of Electrolysis, by André Brochet .....                                              | 279  |
| The Separation of Iron from Nickel and Cobalt by Lead Oxide (Field's Method), by T. H. Laby .....                                               | 280  |
| Report on the Determination of Nitrogen, by F. W. Morse .....                                                                                   | 282  |
| PHYSICAL SOCIETY .....                                                                                                                          | 284  |
| NOTICES OF BOOKS .....                                                                                                                          | 285  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                     | 286  |

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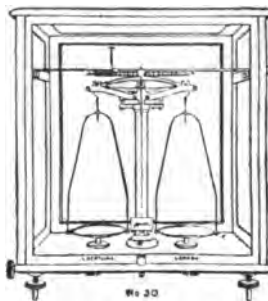
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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2324.

## PREPARATION OF SAMARIA AND ATOMIC WEIGHT OF SAMARIUM.

By G. URBAIN and H. LACOMBE.

DEMARÇAY has already prepared and described pure samaria (*Comptes Rendus*, cxxx., 1185). He has also satisfied himself that no intermediate element exists between neodymium and samarium. Further, he found that pure samarium gives more variation in the absorption than in the spark spectrum.

However, in his method there are eight to ten fractions between the samarium and the europium, and he could scarcely affirm on this evidence that no unknown earth exists between these two elements, the very characteristics of europium being of such a nature as to warrant its being of a complex nature.

We mentioned the principle of our method in a preceding communication (*Comptes Rendus*, cxxxvii., 792; cxxxviii., 84; *CHEMICAL NEWS*, lxxxviii., 295; lxxxix., 52); and, on further development of this, we have been able to actually separate completely from a mixture of rare earths pure samarium oxide.

been explained in our previous communication on europium. Particularly, we have allowed for the presence of free sulphuric acid in our sulphates, and so we have precipitated and washed with alcohol before crystallising from water until the aqueous solution is absolutely neutral to litmus.

On the other hand, the consecutive estimations of water are controlled by the sulphuric acid, and if the crystals only contain octohydrates we can obtain between the calculated atomic weights—either by the estimation of water, or by the estimation of sulphuric anhydride—a remarkable concordance, which is shown by the following measurements:—

|                                                 | First<br>measure-<br>ment. | Second<br>measure-<br>ment. | Third<br>measure-<br>ment. | Mean. |
|-------------------------------------------------|----------------------------|-----------------------------|----------------------------|-------|
| Hydrated sulphate ..                            | 2'5107                     | 3'2200                      | 2'8382                     |       |
| Anhydrous sulphate ..                           | 2'0172                     | 2'5872                      | 2'2804                     |       |
| Oxide .. .. .                                   | 1'1948                     | 1'5324                      | 1'3508                     |       |
| H <sub>2</sub> O, per cent .. ..                | 19'655                     | 19'652                      | 19'653                     |       |
| SO <sub>3</sub> , per cent .. ..                | 32'755                     | 32'757                      | 32'753                     |       |
| Sa <sub>2</sub> O <sub>3</sub> , per cent .. .. | 47'588                     | 47'590                      | 47'593                     |       |

Atomic weight deduced  
from the transforma-  
tion of—

|                                                                                                                                    |        |        |        |         |
|------------------------------------------------------------------------------------------------------------------------------------|--------|--------|--------|---------|
| Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> +8H <sub>2</sub> O into<br>Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .. .. . | 150'30 | 150'37 | 150'35 | 150'340 |
| Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> into Sa <sub>2</sub> O <sub>3</sub> ..                                             | 150'34 | 150'33 | 150'37 | 150'346 |
| Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> +8H <sub>2</sub> O into<br>Sa <sub>2</sub> O <sub>3</sub> .. .. .                  | 150'33 | 150'34 | 150'37 | 150'346 |

|                                                                                                                         | Samarium extracted from<br>gadolinite. |         |        | Samarium extracted from earths<br>rich in yttria (monazite sands). |         |        | Samarium extracted from our first<br>material containing europium. |         |        |
|-------------------------------------------------------------------------------------------------------------------------|----------------------------------------|---------|--------|--------------------------------------------------------------------|---------|--------|--------------------------------------------------------------------|---------|--------|
|                                                                                                                         | Heads.                                 | Middle. | Tails. | Heads.                                                             | Middle. | Tails. | Heads.                                                             | Middle. | Tails. |
| Hydrated sulphate (grms.) ..                                                                                            | 1'0499                                 | 1'2898  | 1'3650 | 1'7992                                                             | 1'8636  | 0'8407 | 2'5107                                                             | 3'1171  | 2'9425 |
| Anhydrous sulphate (grms.) ..                                                                                           | 0'8435                                 | 1'0362  | 1'0969 | 1'4453                                                             | 1'4977  | 0'6749 | 2'0172                                                             | 2'5045  | 2'3635 |
| Oxide (grms.) .. .. .                                                                                                   | 0'4996                                 | 0'6137  | 0'6497 | 0'8557                                                             | 0'8873  | 0'4001 | 1'1948                                                             | 1'4840  | 1'4004 |
| H <sub>2</sub> O, per cent .. .. .                                                                                      | 19'659                                 | 19'661  | 19'641 | 19'669                                                             | 19'634  | 19'721 | 19'655                                                             | 19'652  | 19'677 |
| SO <sub>3</sub> , per cent .. .. .                                                                                      | 32'755                                 | 32'757  | 32'761 | 32'770                                                             | 32'753  | 32'687 | 32'755                                                             | 32'738  | 32'730 |
| Sa <sub>2</sub> O <sub>3</sub> , per cent .. .. .                                                                       | 47'585                                 | 47'597  | 47'597 | 47'560                                                             | 47'612  | 47'591 | 47'588                                                             | 47'608  | 47'592 |
| Atomic weight deduced from the<br>transformation of—                                                                    |                                        |         |        |                                                                    |         |        |                                                                    |         |        |
| Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> +8H <sub>2</sub> O into Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> | 150'24                                 | 150'19  | 150'58 | 150'04                                                             | 150'71  | 149'08 | 150'30                                                             | 150'36  | 149'91 |
| Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> into Sa <sub>2</sub> O <sub>3</sub> .. ..                               | 150'33                                 | 150'30  | 150'34 | 150'16                                                             | 150'44  | 150'72 | 150'34                                                             | 150'50  | 150'49 |
| Sa <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> +8H <sub>2</sub> O into Sa <sub>2</sub> O <sub>3</sub> ..               | 150'31                                 | 150'28  | 150'13 | 150'13                                                             | 150'50  | 150'35 | 150'33                                                             | 150'47  | 150'36 |

By several similar treatments we have collected about 150 grms. of rigorously pure samarium oxide. We also verified the different fractions, and found that they possessed absolutely the same characteristic spectra. Besides this, we thought it necessary to discover whether the atomic weight of the samarium remained constant at the end of another of these fractionations.

The determinations given in the accompanying table are the first which have been effected on samarium absolutely free from europium and gadolinium.

Our numbers, which differ very slightly from those of Clève, are higher than those which Demarçay obtained by the synthesis of the sulphate—a method in which he recognised decided irregularities.

In this latter method the majority of errors tend to diminish the atomic weight. On the other hand, the difference between the atomic weight of samarium and europium is so small that it would not explain the discrepancy between Clève's and Demarçay's numbers (150 and 148), because, in this case, Clève's samaria should not contain more than two-thirds of its weight of europium oxide, on account of the extreme rarity of this element. Further, if our numbers are slightly inferior to Clève's, this fact again points to the insufficiency of the method of synthesis by the sulphates employed by all Swedish chemists. Our method allows of very little error; the principle has

We conclude from these measurements that, admitting O=16, the atomic weight of samarium does not differ sensibly from the number 150'34. — *Comptes Rendus*, cxxxviii., No. 19.

## A NEW METHOD FOR THE FRACTIONATION OF THE CERIC EARTHS.

By H. LACOMBE.

It being admitted that no method exists for quantitative separation in the rare-earth group, we can only have recourse to methods of fractionation to effect the separation of the simple elements forming this important series.

Of all the methods employed, fractional crystallisation of the salts in approved solvents gives the best results up to the present. This general method is the more successful as the salts used have the greatest differences in solubility from one element to the next. Now, these differences are greater, as a rule, in the case of the double salts than with the simple ones.

It was in this manner that Auer von Welsbach in 1885 (*Mon. f. Chem.*, vol. vi., p. 477), by fractionating the double ammoniacal nitrates of the earths of didymium,



succeeded in separating the lanthanum from the didymium and in splitting up this latter body into two component parts, praseodymium with green salts and neodymium with pink salts. However, a time comes, while carrying out this separation, when the fractions not sensibly containing lanthanum give syrupy solutions uncrystallisable even in concentrated nitric acid. In such a case Auer recommended the preliminary elimination of the yttric earths, which accumulate in the latter portions by fusion with nitrates, than to fractionate the raw, impure neodymium obtained in the form of the double sodic salt after having mixed it with a certain proportion of pure lanthanum, of which the action is to carry down the last traces of the praseodymium. Considering the relatively great difficulties met with in the preparation of pure lanthanum, the addition of this body to the neodymium appears to be rather audacious, and chemists who have prepared the rare earths in a state of purity are not, as a rule, very much disposed to re-mix the substances obtained at the cost of so much time and labour.

More recently, Demarçay (*Comptes Rendus*, vol. cxxx., p. 1019) has given an excellent method for the separation of samarium from neodymium by fractional crystallisation of the double magnesian nitrates in concentrated nitric acid. This method enables us easily to get rid of the properly called ceric earths from the yttric earths, from yttrium to samarium included. It can be substituted with great advantage for the fusion methods described by Auer. However, the double nitrates of magnesium do not easily lend themselves to the separation of the components of the old didymium.

Consequently, in the hope of obtaining an easier separation, I have endeavoured to find a salt whose solubility was less than that of the double alkaline nitrates, and greater than that of the double nitrates of magnesium. The double nitrate of manganese, corresponding to the type  $2\text{Di}(\text{NO}_3)_3 + 3\text{Mn}(\text{NO}_3)_2 + 24\text{H}_2\text{O}$ , is the one that has given me the best results. My experiments have been carried out successively on earths rich in neodymium and on earths rich in praseodymium.

In both cases the double nitrates of didymium and manganese are best fractionated in nitric acid of density 1.3.

The proportion of mother-liquors ought to be kept practically constant, and always in small quantity compared with the crystals. During the concentrations we sometimes observe a slight decomposition of the nitrate of manganese with the formation of the brown peroxide. In such a case it suffices to add a few drops of fuming nitric acid to the warm liquor, and it immediately becomes clear by the reduction of the peroxide of manganese, which is then dissolved instantly. To prevent supersaturation and to cause a slow crystallisation, it is as well to start the crystals with a nucleus of the double nitrate of bismuth and manganese, a salt isomorphous with those of the rare earths.

**Earths Rich in Neodymium.**—In twenty-four hours I obtained three consecutive fractions. The mother-liquors of Fraction 3 gave an intense absorption spectrum of samarium, and very slightly the band  $\lambda = 525$ , which is characteristic of neodymium. The samarium obtained in this manner was thus fairly pure. The proportion of neodymium, which can be detected by absorption under these conditions, being about a thousandth.

This separation of samarium is still more rapid and more decided than that proposed by Demarçay. It enables us to separate samarium from neodymium as easily as the use of the double nitrates enables us to separate nickel and cobalt, for example.

After having eliminated the samarium in this manner, I continued the fractionation so as to obtain eight consecutive fractions. The first six showed the praseodymium bands very distinctly. The crystals from the seventh fraction, dissolved in their water of crystallisation and examined through a thickness of 6 centimetres, hardly showed the characteristic blue bands of this element, while the very faint blue bands of neodymium were very distinct.

The eighth fraction was free from praseodymium, and its oxide was sky-blue in colour.

**Earths Rich in Praseodymium.**—This fractionation was continued until ten fractions were obtained. The first was decidedly green. The colour of the praseodymium masks that of the manganese. The following fractions had an undecided tint, and the last had the characteristic carmine colour of these salts of neodymium. Fraction 1 showed no trace of neodymium bands by absorption. The fractionation was continued by successively eliminating the fractions at one end containing no more neodymium, and the fractions at the other end containing no more praseodymium. After three hundred series of crystallisations, the proportion of intermediate fractions was reduced to such a point that the fractionation could no longer be carried out. This is the first time to my knowledge that in a separation of neodymium and praseodymium, the proportion of the intermediate fractions has been brought to a negligible point compared with the quantities of the elements separated at the two extremities of the fractionation.

The small quantity of lanthanum present in my earths was all accumulated in the first fractions.

I have further applied this method to earths containing much lanthanum and some fair amount of didymium; with five fractions only I completely eliminated the didymium from the first three fractions.

I am not prepared to pronounce very definitely as to the value of my method from the point of view of the separation of praseodymium and lanthanum. Still, I have observed that it can be effected.

To sum up, the method I propose enables us to eliminate samarium still more rapidly than by Demarçay's method, and it has incontestable advantages over that of Auer, so far as the separation of neodymium and praseodymium is concerned, since it enables us to reduce the portions intermediate between these two elements to very small quantities in proportion to the elements obtained in a state of purity.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 10.

## INFLUENCE OF THE PHYSICAL NATURE OF THE ANODE ON THE CONSTITUTION OF ELECTROLYTIC PEROXIDE OF LEAD. APPLICATION TO ANALYSIS.

By A. HOLLARD.

WE have already shown (*Bull. Soc. Chim.*, vol. xxix., p. 151) that the peroxide of lead deposited on a platinised platinum anode, in a saline solution of lead traversed by an electric current, is always accompanied by a notable amount of superoxides. In fact, the analytic factor by which the weight of peroxide deposited must be multiplied, to obtain the corresponding weight of lead, is lower than the ratio  $\frac{\text{Pb}}{\text{PbO}_2} = 0.866$  of the molecular weights of lead

and of its binoxide. This analytic factor varies, according to a perfectly regular curve, with the concentration of the lead in the bath; the extreme values we have measured are 0.740 and 0.861, which correspond to 0.1 and 10 grms. of lead for a volume of 300 c.c. of the bath.

We have repeated these experiments with the same bath and the same strength of current, but substituting for the platinised platinum anode an anode of platinum roughened by a sand-blast. The analytic factor then takes a constant value equal to 0.853, no matter what the concentration of the bath may be. This figure being lower than 0.866 indicates the presence of superoxides accompanying the  $\text{PbO}_2$ .

Thus with an anode of platinised platinum, the phenomena of superoxidation are very strongly marked with weak solutions of lead and only slightly pronounced with strong concentrations. On the other hand, with an anode of depolished platinum, the phenomena of superoxidation

remain constant no matter what the concentration may be. Again, the deposits behave differently; with platinised platinum the deposits are very compact no matter what quantity of lead there may be; with depolished platinum we can hardly deposit more than 1 grm. of lead in the form of peroxide.

However, this latter quantity of lead is generally sufficient in analysis, therefore we recommend the use of depolished platinum for the analysis of lead rather than use platinised platinum which necessitates the use of a curve to find the analytic factor.

We cannot use polished platinum, as the peroxide of lead will not stick to it.

The following tables give a summary of the experiments which gave us the factor 0.853 with depolished platinum. Our electrode of platinum gauze served as an anode in these experiments.

The experimental conditions of Table I. are identically the same as those described already (*Bull. Soc. Chim.*, vol. xxix., p. 151); the lead was in the form of nitrate dissolved in an excess of nitric acid.

The experiments in Table II. were made for the purpose of measuring the analytic factor when the lead is in the form of sulphate. For this purpose the sulphate of lead is dissolved in nitrate of ammonia and nitric acid, or, to be more exact, in the following mixture:—40 c.c. of ammonia (density = 0.924) and 67 c.c. of nitric acid (density = 1.33). As in Table I. the bath contained 10 grms. of copper in the state of nitrate.

#### I. Solution of Nitrate of Lead.

| Lead weighed. | Factor, $\frac{\text{Pb}}{\text{PbOx}}$ |
|---------------|-----------------------------------------|
| 1.0004        | 0.8555                                  |
| 0.5001        | 0.8533                                  |
| 0.2005        | 0.8543                                  |
| 0.1006        | 0.8532                                  |
| 0.0698        | 0.8477                                  |
| 0.0500        | 0.8532                                  |
| 0.0301        | 0.8551                                  |
| 0.0103        | 0.8483                                  |

The mean of these results is 0.853.

#### II. Solution of Sulphate of Lead.

|        |        |
|--------|--------|
| 0.1002 | 0.8579 |
| 0.2999 | 0.8549 |
| 0.5001 | 0.8507 |
| 0.9996 | 0.8485 |

Mean = 0.853.

—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 5.

### THE FORMATION OF BASIC SALTS OF COPPER UNDER THE INFLUENCE OF ELECTROLYSIS.

By ANDRÉ BROCHET.

WHEN we electrolyse a solution of chlorate of copper, using an anode of this metal, we notice the formation of basic salts containing a certain amount of chloride, as does also the solution. The metallic deposit obtained at the negative pole is a bad one, without cohesion and also containing chloride.

The anode is eaten away in the same manner as in the electrolysis of chlorate of potassium; it is pitted at numerous points, and eventually becomes perforated, while other parts remain entirely unattacked. By continuing the operation for a sufficiently long time the anode is transformed into a sort of lace work.

These experiments were carried out in the apparatus already described in the paper on the action of copper on chloric acid, some at the ordinary temperature, and others at the temperature of the water-bath.

The conditions were the same as in the case of the

electrolysis of chloric acid; the intensity of the current was 4 ampères for a metallic strip of 1 square decimetre. The back pressure was zero, naturally, and the electromotive force about 2 volts. The solution of chlorate of copper used contained half a grm.-molecule per litre.

The pale blue precipitate formed, and of which a certain quantity adheres to the anode, is whiter in the lower parts. If we filter the solution on the one hand, and on the other hand rapidly wash the anode with a dilute solution of nitric acid (60 c.c. per litre), having no sensible action on the copper and consequently no reducing power on the chlorate, we observe on estimation:—

1. That the amount of copper at the anode is greater than that deposited in the voltameter.
2. That the precipitate and the solution contain a certain quantity of chlorine in the form of chloride.
3. That the amount of chloride formed is greater than that corresponding to the reduction of the chlorate of copper, admitting that the cathodic reduction takes place in this manner, which, however, is not the case since there is a deposit of copper difficult to estimate.

The ratio of the weight of copper to the weight of chlorine in the form of chloride is greater than in the action of chloric acid; this is because of the precipitation of the copper, and consequently little or no reduction of the chlorate.

The electrolysis of chlorate of copper, complicated in appearance, becomes very simple when we know the action of copper on chloric acid and chlorate of copper.

In a previous paper I examined the first of these reactions, and have already referred to the second, which, however, requires some complementary explanation.

The reduction of chlorate of copper by copper, either in a strip or as turnings, always takes place in the same manner. All that varies is the speed of the reaction. In every case the copper becomes covered with a layer of cuprous hydrate. Apparently this product is formed simultaneously with cupric chloride. The cuprous hydrate becomes oxidised at the expense of the chlorate.

The cupric oxide thus formed unites with the chloride to form a basic salt, but as it is in great excess it also precipitates a certain quantity of chlorate, forming a mixed basic compound. We observe, in fact, that at the commencement of the reaction there is a certain quantity of chloride in solution, and that after some time this salt has almost entirely disappeared.

The compound obtained by electrolysis is formed as before of chlorate and chloride, as is easy to show.

The powders obtained by the two processes, dissolved in nitric acid, give an abundant precipitate with nitrate of silver; both give oxidised compounds of chlorine with sulphuric acid, but while the salts obtained by electrolysis decrepitate with violence, the others have only a very slightly energetic action.

All these salts have not the same composition, but in each the ratio Cu : Cl is practically constant and corresponds to the formula given for the basic chloride of copper.

The different products obtained by the action of copper on the chlorate, with or without electrolysis, thus answers to the formula— $\text{Cu}(\text{A})_{2.3}\text{Cu}(\text{OH})_2$ , in which Cl and  $\text{ClO}_3$  can be substituted one for the other in  $\text{A}_2$ . But when the salt obtained by the direct action of copper on the chlorate contains principally chloride, which explains the absence of this latter salt in solution, as we have seen, the electrolytic product contains almost as much chlorate as chloride; on the other hand, the solution is then richer in chloride. These differences have to do, perhaps, with the greater or less richness of the solution in chlorate; they explain the difference in the action of concentrated sulphuric acid.

**Conclusions.**—The action of copper on chlorate of copper is identical with that which it exercises on chloric acid. Both one and the other are produced successively

when we react with the metal on chloric acid with or without the help of electrolysis.

As soon as the acid is saturated the chlorate comes into play, but the speed of the reaction is much less. There is formed on the one hand the cupric salt, on the other the cuprous salt, now the chloride, now the hydrate; in certain cases, perhaps even always, a mixture of the two.

These salts of copper became oxidised at the expense of the chlorate; according to the acidity of the solution the hydrate dissolves or remains precipitated, carrying with it the chlorate or the chloride in the form of mixed basic salts. It follows that the ratio of the dissolved copper to the copper precipitated in the voltameter varies from 1.5 to 2 according to the conditions.—*Bull. Soc. Chim.*, Series 1, vol. xxxi., No. 6.

### THE SEPARATION OF IRON FROM NICKEL AND COBALT BY LEAD OXIDE (FIELD'S METHOD).\*

By T. H. LABY,  
Junior Demonstrator of Chemistry, University of Sydney.

#### REVIEW OF METHODS FOR THE SEPARATION OF IRON FROM NICKEL AND COBALT.

##### 1. Ammonium Hydrate and Chloride.

THE precipitation of ferric salts by ammonium hydrate in the presence of ammonium chloride gives an incomplete separation—according to Moore an absolutely worthless one (*CHEMICAL NEWS*, 1892, lxx., p. 75). No experimental work on the re-precipitations necessary for accuracy was found. Fresenius states three to be necessary. Baumhauer is said to have found that ferric hydrate may occlude 27 per cent of nickel and 48 per cent of cobalt (*Archives fur Néerlandaises*, 1870, vol. vi., reference not verified).

##### 2. Ammonium Carbonate (Schwarzenberg, *Liebig's Annalen*, xlvii., p. 216; also *Chem. Gaz.*, 1856).

To the solution of the chlorides, containing ammonium chloride equal to twenty times the weight of the oxide of nickel present, ammonium carbonate is added to a point specified in Schwarzenberg's paper, and quoted by Fresenius. The success of the method depends on striking a point difficult to ascertain. The method, however, is recommended by Fresenius and others. The writer, in an analysis of a meteoric iron, found it to be a long and tedious separation; and the amount of nickel found being lower than by Field's method, partly owing to a slight loss in the precipitation of the nickel as sulphide and subsequent electrolysis. In this and the previous method the presence of much ammonium chloride makes the solution unsuitable for the immediate determination of the nickel by electrolysis. The solubility of the sulphide in excess of ammonium sulphide may make the sulphide precipitation inaccurate (Terri, *Chem. Gaz.*, 1857).

##### 3. Basic Acetate.

Kessler uses a solution containing 1 grm. of ferric iron per 500 c.c., and adds 1 grm. each of acetic acid and sodium acetate (*Ber.*, also *CHEMICAL NEWS*, xxvii., 14). The presence of much of the latter caused the iron to carry down manganese. Meineke adds a small quantity of acetic acid and sodium acetate to a neutral solution, and gives a short rapid boil (*Zeit. Angew. Chem.*, 1888, [2], xix., 232). Mackintosh believes ammonio-cobalt bases are formed which are peroxidised, finding five or six precipitations necessary; and that cobalt is more difficult to separate than nickel (*School of Mines Quarterly*, July, 1887). Moore says four precipitations are necessary (*CHEMICAL NEWS*, 1892, lxx., 75). Brearley, who has done much to elucidate

the acetate process, found, for a ratio first determined by Kessler, that the solution produced by carefully adding sodium or ammonium carbonate to a cool ferric chloride solution till incipient precipitation, contains then about seven-eighths of the ferric chloride as "dissolved hydrate." Brearley (*CHEMICAL NEWS*, 1899, lxxix., 194; 1897, lxxvi., 210) adds sodium or ammonium carbonate to the cool ferric and nickel chlorides, &c., till a slight permanent precipitate forms, 10 c.c. of 5 E. (or 5 normal) acetic acid, water to about a litre, then 10—12 c.c. of 0.36 E. ammonium or sodium acetate for each grm. of iron present, and raises to the boiling-point. He showed the separation is for total dissolved hydrate solutions most nearly complete when this small amount of acetate is used. Either the sodium or ammonium salts may be used. An increase of acetic acid tends to improve the separation, while an increase of acetate retards it; for example, taking 1.0 grm. of iron, 0.1 grm. of nickel, 10 c.c. of 5 E. acetic, and 10 c.c. of 4.4 E. ammonium acetate (122 c.c. of 0.36 E.) only 0.0884 grm. of nickel was recovered. His results show the acetate may be better added to a boiling solution than a cold one, though this is opposed to the usual custom. Contrary to the opinions of Moore and Mackintosh, his results would show cobalt to be more readily separated from iron than nickel is. The acetate and acetic acid mentioned first above, was insufficient for 1.0 grm. of iron in the presence of 0.1 grm. of aluminium, but 60 c.c. were necessary, and only 98 per cent of the nickel was then recovered. Nickel acetate though soluble in ammonium chloride is insoluble in acetic acid. Ferric acetate is decomposed at 100° C. into the hydrate and acetic acid.

##### 4. Phosphate Method.

Cheney and Richards (*Am. Journ. Sci.*, [3], xiv., 178—181, and *CHEMICAL NEWS*, 1877) precipitated the ferric iron by sodium phosphate in the presence of acetic acid. With more than 3 per cent of nickel re-precipitation was necessary, 99 per cent of the nickel was recovered. Moore, nine years later, apparently independently, published the same method recommending two precipitations for accurate work, but gave no test analysis (*CHEMICAL NEWS*, 1886, liv., 309).

##### 5. Electrolysis.

Le Roy (*Comptes Rendus*, cxii., 772—3) deposits the nickel, cobalt, and iron from an ammonium citrate, sulphate, and hydrate solution, replaces it by a sulphate and hydrate one, and then reversing the current re-dissolves and re-precipitates the nickel and cobalt, while the iron goes into suspension as ferric hydrate. No confirmatory results are given. Engels states that nickel may be deposited free from iron if the latter is thoroughly oxidised by hydrogen peroxide (*Journ. Chem. Soc.*, Abs., 1898, ii., 192; "Rundschau," 1896, 20—24). Foerster finds iron to be deposited (*Journ. Chem. Soc.*, 1898, ii., 228). Ducru obtains the nickel, cobalt, and iron (ferric) as sulphates, and electrolyses in the presence of the ferric hydrate precipitated by the free ammonia present (*Bull. Soc. Chim.*, 1897, [3], xvii.). An irregular amount of iron, about 0.5 per cent of the total present, is deposited with the nickel, partially insoluble in concentrated hydrochloric acid. Using the usual sulphate solution Neumann finds (*Chem. Zeit.*, 1898, [72], xxxii., 731—732), contrary to Engels and Ducru, that with varying conditions of time and strength of current a quite irregular amount of iron is deposited (if more than 0.1 grm. of iron as ferric hydrate is present), which is tedious and inaccurate to determine volumetrically.

##### 6. The Ether Process.

The solubility of ferric chloride, and insolubility of man- ganous, nickelous, chromic, and aluminium chlorides in ether in the presence of hydrochloric acid, was pointed out by Rothe in 1892 (Abstract in *CHEMICAL NEWS*, 1892, lxvi., 182; "Mitth. a. d. Kgl. Tech. Versuchs.," Anstalten, Berlin), who used it as a means of separating iron from cobalt, copper, and these metals. Copper and cobalt partly

\* Read before the Royal Society of N.S. Wales, September 2, 1903. From the *Journ. and Proc. Royal Society of N.S. W.*, xxxvii., p. 157.

in solution in the ether may be removed by shaking out the ether with 5·8 E. (S.G. 1·104) hydrochloric acid. A modification of this is the solution of the chlorides (0·4 grm.) in a minimum of water, and 10 c.c. of concentrated hydrochloric acid, the addition of 10 c.c. of ether, and the passing into it of hydrochloric acid gas at 0° C. (Pinerua, *CHEMICAL NEWS*, 1897, lxxv., 193). The nickel is precipitated as the yellow chloride; the iron and cobalt are in solution. Adaptations for technical work, nickel steel and ores, have been published. Langmuir (*Journ. Am. Chem. Soc.*, xxii., 1900) precipitates the iron with ammonium hydrate (the sulphuric acid present in the case of some ores, &c., interferes with the ether extraction) before applying the separation of the chlorides. Acetone may be added to the ether (Norris, *Journ. Soc. Chem. Ind.*, 1901, xx., 6). The separation has been elucidated by Speller (*CHEMICAL NEWS*, lxxxiii., 124; Sargent, *Journ. Am. Chem. Soc.*, October, 1899, 854). A minimum of hydrochloric acid of strictly 1·1—1·11 S.G. should be used to dissolve the chlorides, 5 c.c. of ether being added per 0·1 grm. of Fe. Two separations are made. The ether extraction process has yet to be tested as thoroughly as Kern tested it for iron from uranium (*Journ. Am. Chem. Soc.*, 1902, xxiii., 10). A slight alteration of the concentration of the reagents affects considerably the behaviour of the cobalt.

#### 7. Field's Process.

Field proposed the method examined in the following experiments (*CHEMICAL NEWS*, 1859, i., 4). It is based on the precipitation by litharge of iron as ferric hydrate from a neutral solution of the nitrates of iron, nickel, and cobalt. He gave two results of test analyses in support for nickel, but none for cobalt. Cheney and Richards (*CHEMICAL NEWS*, 1877, xxxvi., 161) found "by far the best success was obtained in the use of the method given by Field," but Moore says "Field's process . . . cannot be recommended" (*CHEMICAL NEWS*, 1886, liv., 306).

#### Experimental—Advantages of Field's Method.

An inquiry into the accuracy of Field's method was made, as it had distinct advantages over methods commonly in use, viz., a single precipitation of the iron, and the absence, after the removal of the added lead, of all reagents such as ammonium or sodium salts. When combined with the electrolytic determination of nickel and cobalt the method becomes more rapid than, say, a double precipitation of the iron by the basic acetate process and the precipitation of the nickel and cobalt as sulphides. The electrolytic determination, which is highly accurate and convenient, cannot be readily combined with the acetate process, as some experimenters find that ammonium chloride is detrimental to the deposition of the nickel and cobalt ("Electrolytic Methods of Analysis," Neumann; but compare Oettel, *Zeitschr. f. Electrochem.*, 1894, i., 194).

#### Test Analyses.

In the following experiments, Table I., the amount of iron mentioned was taken in the form of a solution of the nitrate together with cobalt or nickel nitrates, and evaporated in a porcelain dish on a water-bath to dryness. It was found that the iron is precipitated as ferric hydrate most readily when the evaporation is stopped just before dryness, and the formation of a very basic nitrate of iron is avoided. The residue from the evaporation was diluted, brought to the boil, and the lead monoxide added while the solution was boiling either in the dish or preferably in a conical flask. On the addition of sufficient lead oxide (six times the weight of the iron present) ferric hydrate separated out in a form which was readily filtered; but often the washing was most tedious, and some of the precipitate passed through the filter-paper. In the first experiments hot water was used, but it was found better, as would be expected, to wash with a solution of some salt, lead nitrate being the one used. The presence of a small quantity of ferric hydrate does not interfere with the accuracy of the

electrolytic deposition of nickel and cobalt. The filtrate contains the nitrates of lead and cobalt or nickel. This lead was removed in some cases by evaporating the filtrate nearly to dryness, adding an excess of sulphuric acid, 50 c.c. of 5 E. to every 1—2 grms. of lead present, decomposing the nitrates, diluting, and finally filtering off the lead sulphate. In the case of experiments 5, 6, 8, and 9, Table I., 6, 7, 8, and 9, Table II., the lead sulphate precipitated from the boiling solution by 50 c.c. of 5 E.  $H_2SO_4$  was filtered off without expelling the nitric acid. After adding an excess of ammonium hydrate these filtrates were electrolysed.

Standard Solutions of the nitrates of iron, nickel, and cobalt were prepared:—

*Nitrate of iron* from iron wire containing 0·07 per cent of carbon.

*Nitrate of Cobalt*: 25 grms. of cobalt sulphate and 50 grms. of ammonium sulphate were dissolved in water, made very slightly acid, sulphuretted hydrogen passed through the solution, and the resulting precipitate removed. The filtrate was made alkaline with ammonium hydrate, and boiled for a long time in order to remove the iron as ferric hydrate. On electrolysis this solution gave a dull grey firmly adherent deposit, which was weighed, dissolved in nitric acid, and the solution made to a known volume. This purification would give a cobalt nitrate sufficiently free from all impurities, excepting nickel.

TABLE I.—Cobalt.

| No. | Iron taken.<br>Grm. | Cobalt taken. | Cobalt found. | Error.  |                                                    |
|-----|---------------------|---------------|---------------|---------|----------------------------------------------------|
| 1.  | 0·004               | 0·2289        | 0·2293        | +0·0004 |                                                    |
| 2.  | 0·004               | 0·2289        | 0·2290        | +0·0001 |                                                    |
| 3.  | none                | 0·2289        | 0·2287        | -0·0002 |                                                    |
| 4.  | 0·2                 | 0·2289        | 0·2253        | -0·0036 | Cobalt not completely removed by electrolysis.     |
| 5.  | 0·2                 | 0·2389        | 0·2266        | -0·0023 |                                                    |
| 6.  | 0·2                 | 0·2289        | 0·2178        | -0·0111 | Cobalt not completely removed by electrolysis. (a) |
| 7.  | 0·2                 | 0·2289        | 0·2221        | -0·0068 |                                                    |
| 8.  | 0·2                 | 0·0229        | 0·0235        | +0·0006 |                                                    |
| 9.  | 0·2                 | 0·0229        | 0·0238        | +0·0009 |                                                    |
| 10. | none                | 0·0458        | 0·0466        | +0·0008 |                                                    |
| 11. | none                | 0·0229        | 0·0235        | +0·0006 |                                                    |

(a) This filtrate, though electrolysed all night and for three hours with 0·8 ampères, still contained cobalt, as was shown by passing sulphuretted hydrogen.

TABLE II.—Nickel.

| No. | Iron taken.<br>Grm. | Nickel taken. | Nickel found. | Error.  |                                                 |
|-----|---------------------|---------------|---------------|---------|-------------------------------------------------|
| 1.  | 0·004               | 0·2149        | 0·2145        | -0·0004 |                                                 |
| 2.  | 0·004               | 0·2149        | 0·2123        | -0·0026 | Ferric hydrate contained nickel.                |
| 3.  | none                | 0·2149        | 0·2147        | -0·0002 |                                                 |
| 4.  | 0·2                 | 0·2149        | 0·2114        | -0·0035 | Iron precipitated by PbO from boiling solution. |
| 5.  | 0·2                 | 0·2149        | 0·2100        | -0·0049 | Iron precipitated by PbO in the cold.           |
| 6.  | 0·2                 | 0·2149        | 0·2122        | -0·0027 | Iron precipitated by PbO from boiling solution. |
| 7.  | none                | 0·2149        | 0·2155        | +0·0006 | Pb added and removed as sulphate.               |
| 8.  | 0·4                 | 0·0430        | 0·0454        | +0·0024 |                                                 |
| 9.  | 0·4                 | 0·0430        | 0·0451        | +0·0001 |                                                 |
| 10. | none                | 0·0430        | 0·0439        | +0·0009 |                                                 |
| 11. | none                | 0·0219        | 0·0224        | +0·0005 |                                                 |

Nitrate of nickel\* was prepared similarly. The flasks and pipettes used were calibrated against calibrated weights. Judging from the greatest difference between the values obtained for any one pipette in this testing, the amounts of cobalt and nickel "taken" are never inaccurate by more than  $\pm 0.0001$  grm.

The equation for the reaction was found to be essentially  $3\text{PbO} + 2\text{Fe}(\text{NO}_3)_3\text{Aq} = 3\text{Pb}(\text{NO}_3)_2 + \text{Fe}_2\text{O}_3\text{Aq}$ . Thus the lead oxide used should be at least six times the weight of iron to be precipitated. In each of the test analyses, slightly more than six times as much lead oxide was used. An excess of 20 c.c. of 17 E. ammonium hydrate was used in each electrolysis.

#### Conclusions.

This method, requiring only one precipitation of the iron, though not so good as claimed by Field, is more accurate for cobalt than the basic acetate process; and as accurate for nickel—about 99 per cent of the latter, and rather more of the former being recovered. It can be very readily combined with the electrolytic determination of nickel and cobalt. Gooch and Medway (*Am. Journ. Sci.*, 1903, xxv. 50) using a rotating cathode, have accurately determined nickel by electrolysis with a current of less than thirty minutes' duration.

The author desires to thank Prof. Liversidge for having given every facility for this research.

### REPORT ON THE DETERMINATION OF NITROGEN.†

By F. W. MORSE.

THE author, in accordance with the vote of the Association last year, undertook the comparison of methods for determining the availability of organic nitrogen. Two methods were recommended by the previous referee for trial, namely, the neutral permanganate method, as modified by Mr. Street, and the alkaline permanganate method as modified by Mr. Jones. The former has been adopted by this Association as a provisional method, while the latter has not been regarded with much favour by the majority of analysts who have tried it.

Since nearly all the principal nitrogenous fertiliser materials have been worked upon in the past by the different methods, the author considered it the best policy this year to confine the work to two typical materials combined with superphosphate in approximately the proportion which might occur in a mixed fertiliser, in order to determine how closely results by different analysts would agree, and to make plainer any defect in the manipulation of the method.

The materials selected were dried blood and cotton-seed meal, because both are known to be highly available to plants, while the alkaline method has always failed to indicate the availability of cotton-seed meal, owing apparently to its carbonaceous matter. The blood was a part of a bag of blood meal for poultry and calves, and contained 13.1 per cent of nitrogen. The cotton-seed meal was a standard brand, and contained 7.49 per cent of nitrogen.

Four lots of material were prepared, containing approximately 3.5 to 4 per cent of nitrogen. The mixtures all contained superphosphate, while the nitrogenous material was arranged as follows:—No. 1, blood; No. 2, cotton-seed meal; No. 3, two-thirds nitrogen from blood, one-third nitrogen from cotton-seed meal; No. 4, one-third nitrogen from blood, two-thirds nitrogen from cotton-seed meal.

All the materials were sifted through the standard sieve, with circular holes 1 m.m. in diameter, and then each lot

TABLE I.—Total Nitrogen.

| Sample No. 1.                                                               | Sample No. 2. | Sample No. 3. | Sample No. 4. | Remarks.                                                 |
|-----------------------------------------------------------------------------|---------------|---------------|---------------|----------------------------------------------------------|
| Per cent.                                                                   | Per cent.     | Per cent.     | Per cent.     |                                                          |
| T. C. Trescott, Washington, D. C. :—                                        |               |               |               |                                                          |
| 3.78                                                                        | 3.30          | 3.82          | 3.73          | Digested two hours.                                      |
| 3.75                                                                        | 3.34          | 3.85          | 3.73          |                                                          |
| 3.98                                                                        | 3.59          | 3.95          | 3.87          | Digested three hours.                                    |
| 4.09                                                                        | 3.60          | 3.95          | 3.85          |                                                          |
| Department of Foods and Feeding, Hatch Experiment Station, Massachusetts :— |               |               |               |                                                          |
| 4.16                                                                        | 3.58          | 4.01          | 3.84          | Digested three hours, Kjeldahl method.                   |
| 4.16                                                                        | —             | 3.99          | 3.84          |                                                          |
| 4.15                                                                        | 3.56          | 3.97          | 3.81          | Digested three hours (Kjeldahl) with sulphate of potash. |
| 4.17                                                                        | 3.64          | 4.07          | 3.96          |                                                          |
| 4.14                                                                        | —             | 4.06          | 3.92          |                                                          |
| 4.14                                                                        | 3.63          | 4.03          | 3.92          |                                                          |
| F. B. Carpenter, Virginia :—                                                |               |               |               |                                                          |
| 4.18                                                                        | 3.59          | 4.06          | 3.97          |                                                          |
| S. H. Sheib, Virginia :—                                                    |               |               |               |                                                          |
| 4.19                                                                        | 3.55          | 4.07          | 3.94          |                                                          |
| L. A. Hill, New Hampshire :—                                                |               |               |               |                                                          |
| 3.90                                                                        | 3.54          | 3.75          | 3.67          | Digested three to four hours.                            |
| 3.98                                                                        | —             | —             | 3.78          |                                                          |
| 4.00                                                                        | 3.54          | 3.89          | 3.73          |                                                          |
| W. W. Braman, New Hampshire :—                                              |               |               |               |                                                          |
| 3.98                                                                        | 3.45          | 3.88          | 3.84          |                                                          |
| 4.03                                                                        | 3.48          | 3.94          | 3.86          |                                                          |
| A. L. Sullivan, New Hampshire :—                                            |               |               |               |                                                          |
| 4.05                                                                        | 3.33          | 3.92          | 3.64          | Gas pressure lower than normal.                          |
| 4.06                                                                        | 3.33          | 3.89          | 3.72          |                                                          |
| J. Bernard Robb, Maryland :—                                                |               |               |               |                                                          |
| 4.03                                                                        | 3.50          | 3.95          | 3.77          | Digested one hour.                                       |
| 4.05                                                                        | 3.47          | 3.93          | 3.79          |                                                          |
| 4.08                                                                        | 3.51          | 3.96          | 3.82          |                                                          |
| C. H. Jones, Vermont :—                                                     |               |               |               |                                                          |
| 4.32                                                                        | 3.69          | 4.04          | 4.04          | Digested two and a-half to three hours.                  |
| 4.35                                                                        | 3.69          | 4.04          | 3.97          |                                                          |
| 4.32                                                                        | 3.76          | 4.11          | 3.97          |                                                          |
| F. W. Woll and Geo. A. Olson, Wisconsin :—                                  |               |               |               |                                                          |
| 4.16                                                                        | 3.58          | 4.01          | 3.93          | Digested from one and a-half to five hours.              |
| 4.19                                                                        | 3.58          | 4.01          | 3.98          |                                                          |
| 4.17                                                                        | 3.60          | 4.08          | 3.95          |                                                          |
| 4.14                                                                        | 3.57          | 4.07          | 3.95          |                                                          |
| —                                                                           | —             | 4.09          | —             |                                                          |
| W. H. Scherffins and A. M. Peter, Kentucky :—                               |               |               |               |                                                          |
| 4.08                                                                        | 3.49          | 3.97          | 3.79          | Digested three hours.                                    |
| 4.07                                                                        | 3.53          | 3.88          | 3.86          |                                                          |
| 4.13                                                                        | 3.54          | 3.95          | 3.86          | Digested five hours.                                     |
| 4.12                                                                        | 3.54          | 3.94          | 3.87          |                                                          |
| W. R. Perkins, Mississippi :—                                               |               |               |               |                                                          |
| 4.02                                                                        | 3.48          | 3.89          | 3.90          |                                                          |

was thoroughly mixed. The blood was apparently coarse, because a large part of it was crystalline rather than fibrous. Two of the analysts re-ground the samples containing it.

Twelve lots of samples were sent to as many different laboratories. Accompanying the samples were sent the following instructions :—

#### Directions for Work on Nitrogen, 1902.

Please determine total nitrogen in each sample by either of the official methods. Last year the referee called attention to the fact that prolonged digestion with the sulphuric acid in the Kjeldahl method raised the amount of nitrogen. Please note the length of time used in each digestion throughout the comparison. Please determine available nitrogen by the neutral permanganate method adopted provisionally at the last convention, and by Jones's alkaline permanganate method, which has so far been regarded

\* Foerster, *Zeit Electrochem.*, 1897, iv., 160—165. C, Si, Cu, Mn entirely absent from electrolytically deposited Ni, Fe, and Co in same proportion as unrefined material.

† Proceedings of the Nineteenth Annual Convention of the Association of Official Agricultural Chemists, held at Washington, D.C.

TABLE II.—*Insoluble Nitrogen (Neutral Permanganate Method).*

| Sample No. 1.                                                              | Sample No. 2. | Sample No. 3. | Sample No. 4. | Remarks.    |
|----------------------------------------------------------------------------|---------------|---------------|---------------|-------------|
| Per cent.                                                                  | Per cent.     | Per cent.     | Per cent.     |             |
| T. C. Trescot, Washington, D.C.:—                                          |               |               |               |             |
| 0.68                                                                       | 0.37          | 0.94          | 0.72          | Washed.     |
| 0.80                                                                       | 0.25          | 0.86          | 0.83          |             |
| 1.90                                                                       | 0.46          | 0.91          | 0.60          | Not washed. |
| 1.78                                                                       | 0.25          | 0.72          | 0.81          |             |
| Department of Foods and Feeding, Hatch Experiment Station, Massachusetts:— |               |               |               |             |
| 1.85                                                                       | 0.40          | 1.22          | 0.93          |             |
| 1.78                                                                       | 0.39          | 1.15          | 0.83          |             |
| S. H. Sheib, Virginia:—                                                    |               |               |               |             |
| 0.65                                                                       | 0.30          | 0.56          | 0.61          |             |
| L. A. Hill, New Hampshire:—                                                |               |               |               |             |
| 0.42                                                                       | 0.31          | 0.70          | 0.84          | Not washed. |
| 0.46                                                                       | 0.56          | 0.78          | 0.80          |             |
| 0.53                                                                       | 0.23          | 0.49          | 0.52          |             |
| 0.61                                                                       | 0.31          | 0.50          | 0.40          |             |
| W. W. Braman, New Hampshire:—                                              |               |               |               |             |
| 0.92                                                                       | 0.56          | 0.87          | 0.77          |             |
| 0.40                                                                       | 0.36          | 0.56          | 0.52          |             |
| A. L. Sullivan, New Hampshire:—                                            |               |               |               |             |
| 1.01                                                                       | 0.38          | 0.67          | 0.50          |             |
| 1.28                                                                       | 0.41          | 0.67          | 0.52          |             |
| C. H. Jones, Vermont:—                                                     |               |               |               |             |
| 1.29                                                                       | 0.35          | 1.06          | 0.87          |             |
| F. W. Woll and Geo. A. Olson, Wisconsin:—                                  |               |               |               |             |
| 0.34                                                                       | 1.17          | 1.04          | 1.52          |             |
| 0.36                                                                       | 1.12          | 0.99          | 1.55          |             |
| W. H. Scherffins and A. M. Peter, Kentucky:—                               |               |               |               |             |
| 0.39                                                                       | 1.20          | 0.97          | 1.51          |             |
| 1.26                                                                       | 0.45          | 1.48          | 0.97          |             |
| 1.47                                                                       | 0.49          | 1.33          | 0.76          |             |
| 1.26                                                                       | 0.36          | 1.53          | 0.83          |             |
| 1.34                                                                       | 0.32          | 1.05          | 0.91          |             |
| W. R. Perkins, Mississippi:—                                               |               |               |               |             |
| 0.59                                                                       | 0.41          | 0.70          | 0.71          |             |
| 0.59                                                                       | 0.41          | 0.68          | 0.64          |             |

unfavourably. The latter method used in accordance with Mr. Jones's practice offers some advantages over the former in the matter of time. The methods are described as follows:—

*The Neutral Permanganate Method.*—Into a 400 c.c. Griffin beaker, low form, weigh an amount of sample containing approximately 0.075 grm. of nitrogen (samples containing material that has been treated with acid should be washed on a 9 c.m. S. S. No. 595 filter to 200 c.c. and transferred, filter and all, to beaker). Digest this with 125 c.c. of permanganate of potash solution (16 grms. of pure  $\text{KMnO}_4$  to 1000 c.c. of water) in a steam- or hot-water bath for thirty minutes. Have the beaker let down well into the steam or hot water, and keep closed with a cover glass, stirring twice at intervals of ten minutes with a glass rod. At the expiration of the time, remove from bath, add 100 c.c. of cold water, and filter through a heavy 15 c.m. folded filter. Wash with cold water, small quantities at a time, till total filtrate amounts to 400 c.c. Dry, and determine nitrogen in residue by Kjeldahl method.

*The Alkaline Permanganate Method.*—Weigh out an amount of sample containing approximately 0.045 grm. of nitrogen, and transfer to a 600 c.c. distillation flask. After connecting with condenser to which the receiver containing standard acid has been attached, digest with 100 c.c. of alkaline permanganate solution (16 grms. of potassium permanganate and 150 grms. of sodium hydrate dissolved in 1000 c.c. of water) for thirty minutes below the boiling-point. Then boil slowly until the distillation is completed.

TABLE III.—*Soluble Nitrogen (Alkaline Permanganate Method).*

| Sample No. 1.                                                               | Sample No. 2. | Sample No. 3. | Sample No. 4. | Remarks.              |
|-----------------------------------------------------------------------------|---------------|---------------|---------------|-----------------------|
| Per cent.                                                                   | Per cent.     | Per cent.     | Per cent.     |                       |
| T. C. Trescot, Washington, D.C. :—                                          |               |               |               |                       |
| 2.46                                                                        | 1.57          | 1.99          | 1.68          |                       |
| 2.32                                                                        | 1.51          | 2.10          | 1.82          |                       |
| Department of Foods and Feeding, Hatch Experiment Station, Massachusetts :— |               |               |               |                       |
| 2.38                                                                        | 1.30          | 1.83          | 1.55          |                       |
| —                                                                           | —             | —             | 1.53          |                       |
| 2.35                                                                        | 1.30          | 1.82          | 1.51          |                       |
| L. A. Hill, New Hampshire :—                                                |               |               |               |                       |
| 2.73                                                                        | 1.82          | 2.31          | 1.82          | } Distilled very low. |
| 2.80                                                                        | 1.96          | 2.38          | 2.13          |                       |
| W. W. Braman, New Hampshire :—                                              |               |               |               |                       |
| 2.99                                                                        | 2.00          | 2.71          | 2.57          | } Distilled very low. |
| 2.98                                                                        | 1.93          | 2.80          | 2.49          |                       |
| A. L. Sullivan, New Hampshire :—                                            |               |               |               |                       |
| 2.90                                                                        | 2.44          | 2.41          | 2.26          | } Distilled very low. |
| 2.97                                                                        | 2.27          | 2.57          | 2.54          |                       |
| J. Bernard Robb, Maryland :—                                                |               |               |               |                       |
| 2.83                                                                        | 1.85          | 2.40          | 2.27          |                       |
| 2.88                                                                        | 1.84          | 2.46          | 2.17          |                       |
| 2.75                                                                        | 1.78          | 2.31          | 2.19          |                       |
| C. H. Jones, Vermont :—                                                     |               |               |               |                       |
| 2.67                                                                        | 1.50          | 2.16          | 1.86          |                       |
| 2.64                                                                        | 1.53          | 2.09          | 1.90          |                       |
| F. W. Woll and Geo. A. Olson, Wisconsin :—                                  |               |               |               |                       |
| 2.22                                                                        | 1.28          | 1.81          | 1.77          |                       |
| 2.33                                                                        | 1.18          | 1.84          | 1.78          |                       |
| 2.27                                                                        | 1.40          | 1.96          | 1.75          |                       |
| 2.17                                                                        | 1.33          | 1.79          | 1.70          |                       |
| W. H. Scherffins and A. M. Peter, Kentucky :—                               |               |               |               |                       |
| 1.64                                                                        | 1.25          | 1.47          | 1.37          |                       |
| 1.79                                                                        | 1.25          | 1.41          | 1.36          |                       |
| 1.83                                                                        | 1.26          | 1.34          | 1.48          |                       |
| W. R. Perkins, Mississippi :—                                               |               |               |               |                       |
| 2.81                                                                        | 1.56          | 2.31          | 2.05          |                       |
| 2.79                                                                        | 1.62          | 2.47          | 2.18          |                       |
| 2.75                                                                        | 1.54          | 2.34          | 2.07          |                       |

TABLE IV.—*Availability of Nitrogen in Neutral Permanganate.*

| Sample No. 1.                                                              | Sample No. 2. | Sample No. 3. | Sample No. 4. | Remarks.              |
|----------------------------------------------------------------------------|---------------|---------------|---------------|-----------------------|
| Per cent.                                                                  | Per cent.     | Per cent.     | Per cent.     |                       |
| T. C. Trescot, Washington, D.C.:—                                          |               |               |               |                       |
| 81.63                                                                      | 91.36         | 77.21         | 80.05         | Washed until neutral. |
| 54.34                                                                      | 90.25         | 79.49         | 81.86         | Unwashed.             |
| Department of Foods and Feeding, Hatch Experiment Station, Massachusetts:— |               |               |               |                       |
| 55.45                                                                      | 89.00         | 69.64         | 76.05         |                       |
| 57.21                                                                      | 89.15         | 71.24         | 78.53         |                       |
| S. H. Sheib, Virginia:—                                                    |               |               |               |                       |
| 84.49                                                                      | 93.52         | 86.24         | 81.97         |                       |
| L. A. Hill, New Hampshire:—                                                |               |               |               |                       |
| 88.97                                                                      | 87.57         | 80.62         | 77.68         | Unwashed samples.     |
| 85.75                                                                      | 92.38         | 86.85         | 81.94         | Washed samples.       |
| W. W. Braman, New Hampshire:—                                              |               |               |               |                       |
| 83.50                                                                      | 87.57         | 81.84         | 83.36         |                       |
| A. L. Sullivan, New Hampshire:—                                            |               |               |               |                       |
| 71.85                                                                      | 88.28         | 82.82         | 81.70         |                       |
| C. H. Jones, Maryland:—                                                    |               |               |               |                       |
| 70.20                                                                      | 90.57         | 73.89         | 79.45         |                       |
| F. W. Woll and Geo. A. Olson, Wisconsin:—                                  |               |               |               |                       |
| 91.36                                                                      | 67.31         | 75.30         | 61.26         |                       |
| W. R. Perkins, Mississippi:—                                               |               |               |               |                       |
| 85.32                                                                      | 88.31         | 82.26         | 82.69         |                       |

TABLE V.—*Availability of Nitrogen in Alkaline Permanganate.*

|                                                                                         | Sample<br>No. 1.<br>Per cent. | Sample<br>No. 2.<br>Per cent. | Sample<br>No. 3.<br>Per cent. | Sample<br>No. 4.<br>Per cent. |
|-----------------------------------------------------------------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| T. C. Trescot, Wash-<br>ington . . . . .                                                | 59'30                         | 42'89                         | 51'64                         | 45'53                         |
| Department of Foods<br>and Feeding, Hatch<br>Experiment Station,<br>Massachusetts . . . | 57'26                         | 56'22                         | 45'45                         | 40'00                         |
|                                                                                         | 56'68                         | 36'04                         | 45'30                         | 39'00                         |
| L. A. Hill, New Hamp-<br>shire . . . . .                                                | 69'17                         | 53'39                         | 61'53                         | 52'95                         |
| W. W. Braman, New<br>Hampshire . . . . .                                                | 74'50                         | 56'64                         | 70'33                         | 65'71                         |
| A. L. Sullivan, New<br>Hampshire . . . . .                                              | 72'34                         | 70'57                         | 63'84                         | 65'21                         |
| J. Bernard Robb, Mary-<br>land . . . . .                                                | 69'63                         | 52'00                         | 60'91                         | 58'15                         |
| C. H. Jones, Vermont<br>F. W. Woll and Geo. A.<br>Olson, Wisconsin . .                  | 61'31                         | 40'88                         | 52'34                         | 47'12                         |
|                                                                                         | 54'19                         | 36'31                         | 45'43                         | 44'30                         |
| W. R. Perkins, Missis-<br>sippi . . . . .                                               | 69'23                         | 45'21                         | 61'01                         | 53'84                         |

Since the samples all contain superphosphates, it will be necessary to wash them as directed for the neutral method. This is not necessary for the alkaline method, as it is intended to be used. Comparisons of washed and unwashed charges will be appreciated, however.

The troublesome features of these methods seem to be filtration in the neutral and distillation in the alkaline method. Suggestions for hastening these steps will be of interest.

#### Results of Analyses.

The methods were copied from the report of last year.

Returns were received from nine laboratories, and included the work of twelve analysts. The accompanying tables give their results in full.

(To be continued).

## PROCEEDINGS OF SOCIETIES.

### PHYSICAL SOCIETY.

Ordinary Meeting, May 27th, 1904.

Mr. J. SWINBURNE, Vice-President, in the Chair.

A PAPER entitled "*The Law of Action between Magnets and its bearing on the Determination of the Horizontal Component of the Earth's Magnetic Field with Unifilar Magnetometers*" was read by Dr. C. CHREE.

Starting with the general formula for the action between two magnets perpendicular to one another, in Lamont's first position, the paper discusses how observations should be combined when the higher terms usually neglected in magnetometer reductions are taken into account. It considers the effect of errors in setting the deflecting magnet or in the values assigned to the graduations of the deflection-bar, and of errors in the deflection-angles, according as observations are made at one, at two, or at three distances. Assuming a simple magnet to be replaceable by poles, and making use of formulæ due originally to Lamont and Børgen, calculations are made as to the size of the several coefficients, P, Q, R, in the deflection formula, for hypothetical values of the pole-distance, in the magnets of the more usual English magnetometer types. These calculated values are compared with the mean values observed at Kew in past years in magnetometers of the several types; and conclusions are drawn as to the mean ratio of the pole-distance to the total length of the magnets. The question of variation in the pole-distance with time is dis-

cussed in connection with recorded mean annual values of the P coefficient for the observatory magnetometers at Kew, Batavia, and Mauritius. The paper concludes with the results of some experiments on the degree of uniformity in the pole-distance of magnets of the same size and pattern. The method consisted essentially in employing two magnets alternately as deflector and deflected in the same magnetometer. Instrumental peculiarities are in this way largely eliminated.

Dr. W. WATSON said that the author had referred in the course of his remarks to a statement which he (Dr. Watson) had made at a former meeting of the Society, to the effect that the limit of accuracy in the setting of the magnet-carriage upon the deflection-bar was 0.1 m.m. Although it might be possible, when working under favourable conditions at an observatory, to make the setting with greater precision, he thought that 0.1 m.m. was as much as could be expected or obtained in ordinary work in the field. He pointed out that the number of settings in a deflection experiment was a safeguard against inaccuracies in individual settings, and that it was probable that there was no source of constant error. He expressed his interest in the author's experiments on pole-distances, and said that when the magnetometers were being designed for the Indian Survey he had expressed the opinion that it might be advantageous to make the coefficient Q equal to zero by choosing suitable pole-distances for the magnets. Probably Dr. Chree's experiments will enable us to settle whether it is sufficiently accurate to make Q as small as possible and work with a value of P only, determined in the ordinary way.

Dr. P. E. SHAW pointed out that brass, which is used for the deflection-bar, has a specially large thermal expansion, and if 50 c.m. of it rises 3° C. the increase in length would be about 25 microns, which Dr. Chree stated to be the error in setting the magnet-carriage on the scale. Possibly some alloy, e.g., some form of invar, would be preferable to brass.

Prof. H. L. CALLENDAR said that some time ago he made some experiments upon the distance between the poles of magnets, and the conclusion he came to was that it was impossible to express the pole-distance as a definite fraction of the length of the magnet. He had found that among other things the pole-distance depended upon the shape of the magnet, the intensity of magnetisation, and the kind of steel of which the magnet was composed. There was also a variation of the pole-distance with time.

Mr. SKINNER said that it might be possible to fix the position of the poles by using magnets similar to those used by Mr. Searle in many of his experiments. These magnets consisted of long thin needles with steel balls at the ends, and it was found that the poles were very close indeed to the centres of the balls.

Dr. CHREE said that the limit of accuracy in the setting of the magnet carriage which he had mentioned, 0.03 m.m., was that which was obtainable when working under the most favourable conditions. With regard to the material of the deflection-bar, he pointed out that a sufficiently accurate temperature correction was always employed to correct for thermal expansion. There was no invar which was sufficiently non-magnetic to be used in accurate work with a magnetometer.

A paper "*On the Ascertained Absence of Effects of Motion through the Æther in relation to the Constitution of Matter on the FitzGerald-Lorentz Hypothesis*," by Prof. J. LARMOR, was taken as read.

In consequence of recent misapprehensions (cf. D. B. Brace, *Phil. Mag.*, March, 1904), the argument on this subject, as given in "*Æther and Matter*" (1900), is briefly re-stated. The absence of effect of convection, to the first order, was demonstrated by Lorentz. Absence of effect to the second order of the ratio of the velocity of convection to that of radiation has now been experimentally established, as regards optical interference with long path, by Michelson; as regards mechanical action on a charged electric con-



denser, by Trouton; as regards double-refraction, by Lord Rayleigh and by Prof. Brace. This suggests strongly a complete correspondence in detail between the material system connected with the earth's motion and the same system at rest in the æther, so that their internal relations are indistinguishable. Theoretically such complete correspondence, up to the second order, exists, involving the FitzGerald-Lorentz shrinkage, provided a purely electrical constitution of matter (as regards its physical relations) is granted, but apparently not otherwise. Thus it is held that these phenomena point consistently in that direction.

A paper "On Coherence and Recoherence," by Dr. P. E. SHAW and Mr. C. A. B. GARRETT, was read by Dr. SHAW.

In a paper in the *Phil. Mag.* (March, 1901), Dr. Shaw described a method of investigating coherence by measuring the forces required to sunder the cohered surfaces. It was there shown that forces of the order of one dyne were required for a copper-copper contact of two single wires. Further, there seemed to be evidence of a change of state at the place of coherence, possibly orientation of the particles at the contact. In the present paper the authors follow the same method of investigation, adducing evidence that coherence can be explained, and only explained, by Lodge's original theory of fusion; and further establishing the after-effect, whether orientation or otherwise, mentioned in the former paper.

When electrical radiation falls on a contact of two wires, under suitable conditions coherence occurs. On applying a small measured force the cohered surfaces separate. On bringing them into contact again at the same places coherence will often, but not always, occur without the incidence of electric radiation. This can be repeated two or three times, but the power to cohere spontaneously soon vanishes. This spontaneous coherence is called recoherence. Like coherence, its presence is indicated by the forces required to sunder the surfaces. Several tables of these results are given.

Allowing that coherence itself is due to fusion, for which the evidence is abundant, recoherence may be due to (1) fusion, (2) molecular or intramolecular change, brought about during coherence by the locally violent surging at the contact.

As to fusion, Prof. J. J. Thomson has suggested that the opposed spikes formed when the surfaces are torn asunder may be so fine as to be fused together by the small circuit current when the surfaces meet again. Without such spikes it is obvious the surfaces would be normal, and no spontaneous coherence could occur. Against the spike theory it is pointed out that recoherence never recurs more than a few times, which indicates some unstable condition of the surfaces. The orientation seen in magnetism can be destroyed by reversals of decreasing currents, by mechanical shock, and by heating. On adopting these methods to destroy recoherence, the authors obtained no definite results.

Recoherence is therefore still unexplained. Two surfaces can be pressed together with small but measurable forces by two methods:—(1) By a boom attached to a galvanometer suspension; a current in the galvanometer regulates the pressure between a wire on the boom and a fixed wire, these two forming the contact. (2) By a light balance of aluminium rendered only just stable; the pressure is regulated by a current in a solenoid, which acts on a magnet attached to one end of the balance. Both these methods are used by the authors to bring the surfaces into contact and to tear them apart again. The forces operating can easily be measured by a subsequent calibration.

Dr. W. WATSON, referring to the fact that when recoherence ceases to occur with a direct current it may be re-established by reversing the current, suggested that it might be due to the effect of the oscillations set up in the circuit at the make and break.

Dr. C. CHREE asked whether the authors could indicate approximately the relative number of cases in which recoherence failed when the current was not reversed, but

occurred when the current was reversed. He suggested that the fragmentary remains of the bridge which the authors described as existing during coherence might sometimes be so shaped as to make it easier for the current to pass in one direction than the other.

Prof. TROUTON asked if recoherence would occur without a current running through the contact. He suggested that one side of the contact might be a point and the other side approximately plane, so that the current would flow more easily in one direction than the other.

Mr. R. S. WHIPPLE asked if the effect depended upon the materials. In the case of a Callendar recorder, with a boom contact, the contact-pieces being made of platinum, it was found that the sensitiveness of the instrument depended upon the direction of the current.

Dr. R. S. CLAY asked if the reversal of the current which established recoherence took place when the substances were in contact.

Dr. P. E. SHAW, in reply to various questions, said that mere reversal of current would not produce the recoherence phenomenon. It only occurs as a sub-permanent effect following coherence, and cannot therefore be attributed to sparking caused by rapid reversals. As to the action of platinum and other materials as contact-surfaces, he found platinum singularly apt at coherence, but recoherence has not been looked for except in a copper-copper contact. Any explanation offered for recoherence must take account of the observed effects, viz., that it is a sub-permanent effect following coherence, is unstable to various influences, and arises most often by reversal of current but sometimes without such reversal.

## NOTICES OF BOOKS

*Refuse Disposal and Power Production.* By W. FRANCIS GOODRICH. With Ninety-eight Illustrations. London: Archibald Constable and Co., Limited. 1904. Pp. 384.

It is only within recent years that the problem of refuse disposal has been tackled seriously and on the large scale. With the increase of sanitary knowledge it has been recognised that the old refuse heaps and pits on the outskirts of towns are admirably adapted for the incubation and, through the action of the wind, the dissemination of all kinds of harmful bacteria. Destruction by burning is the only practicable means for getting rid of this refuse, and though the strict sanitarian condemns the utilisation of the heat produced for the production of power, there can be no doubt that under some circumstance perfect cremation can be effected, and at the same time a great deal of otherwise waste heat utilised for driving such machinery as electric-lighting plant, pumps, &c.

It is true that in these days the commercial aspect or sanitary improvements is very prominent, and it may be that in some cases where the power cannot be utilised, and when it is a question of keeping down either the local council rate or the death-rate, it is the former that receives the greatest consideration, but, on the other hand, in many places that appears to be the last thing that enters into the minds of our local authorities. Still, when both can be done together, and of this the author feels confident, we cannot see any reason why it should not be attempted.

The author has treated the subject in a broad-minded manner, and he has placed sanitation in the forefront as the primary object of the destructor, and he looks upon it as a sanitary necessity, whether the power can be utilised or not.

The book is divided into twenty chapters, the early ones dealing with the old methods of refuse disposal. In Chapter V. we have British destructors described and illustrated, and Chapter VI. is devoted to the cost of labour. Chapters IX., X., and XI. are important ones, and deal with destructors combined with electricity works,

with sewage works, and with water works respectively. The latter combination we must condemn unhesitatingly. It is the constant endeavour of the water engineer, and of the chemist who advises him, to remove to the utmost all possible sources of pollution from the watershed, and we can hardly imagine anything more recklessly unwise than to deliberately cart town refuse into the close vicinity of reservoirs and filter beds day after day throughout the year, under such circumstances that the air must of necessity be loaded with dust, which is one of the most perfect microbe carriers.

Chapters XIII., XIV., and XV. discuss the comparative merits of different practical arrangements for complete combustion and satisfactory steam raising, while the remaining chapters deal with refuse destructors already in operation in London, in the rest of England, in Scotland, Ireland, Wales, and abroad.

There is an index of eight pages.

**Fire and Explosion Risks.** By Dr. VON SCHWARTZ. Translated from the Revised German Edition by CHARLES T. C. SALTER. London: Charles Griffin and Company, Ltd. 1904. Pp. 357.

THIS handbook deals with the detection, investigation, and prevention of dangers arising from fires and explosions of chemo-technical substances and establishments. It is intended for the use of fire insurance officials, fire-brigade officers, members of the legal profession, law officers, councillors, factory inspectors, and factory owners; and the chief endeavour of the author has been to insist on the indispensable aid that chemistry renders to the solution of the important problems arising in connection with the detection of the causes of accidental or premeditated outbreaks of fire.

The work is not designed to form an introduction to the variable practices of fire insurance companies, or a mere repetition of Trade Union regulations for the prevention of accidents, but to present to non-chemists a practical chemical tool, and to facilitate the investigation of the, to them, foreign domain of chemical technology in questions of fire risk and fire prevention.

The book is a large one, and is divided into eleven parts and fifty-nine chapters. The principal headings under which these parts are arranged are the various dangers of fire which may arise under different conditions. For instance, Part I. is on fires and explosions of a general character; Part II., dangers caused by sources of light and heat; Part III., dangers caused by gases; other parts deal with the dangers of various industrial materials, agricultural products, fats, oils, resins, petroleum, mineral oils, alcohols, ether, and other liquids; also dangers produced by metals, oxides, acids or salts, lightning, lighting materials, &c.

The author has dealt with the whole subject in a most comprehensive manner, and has given us a work which is worthy of the time and labour spent in its production. Besides a full table of contents there is a good index, so that any branch of the subject can be turned up in a short time.

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 6th inst., His Grace The Duke of Northumberland, K.G., President, in the Chair. Mr. Richard Bagot, Mr. W. Carrington, Mr. E. G. Fellows, Mr. W. R. Freeman, Mr. R. F. Nicholson, and the Hon. C. A. Parsons, F.R.S., were elected Members. The sincere thanks of the Members were returned to Dr. Andrew Carnegie, who is so intimately associated with the iron and steel industries, for his donation of £1200 to enable Prof. Dewar and Mr. R. A. Hadfield to prosecute their joint investigation on the Physical Properties of Steel and other Alloys at Low Temperatures, and to Dr. Frank McClean for his donation of £100 to the Research Fund of the Royal Institution.

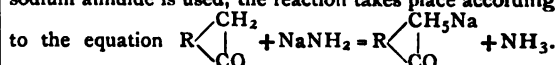
## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 19, May 9, 1904.

**Use of Alternating Currents in Chemistry, and Theory of the Reactions effected by them.**—M. Berthelot.—The use of alternating currents in chemistry has been studied intermittently by several observers. The author summarises these researches, and discusses a theory of the action of such currents.

**A New Method of Preparation of Alcoyl and Alcoylidonic Derivatives of the Cyclic Ketones. Application to the Preparation of Alcoyl Menthones.**—A. Haller.—When certain cyclic ketones are treated with sodium, a sodium derivative and the ketone of the corresponding alcohol is formed. If, instead of the metal itself, sodium amide is used, the reaction takes place according



Reactions of this nature can also be applied to the preparation of the alcoyl menthones.

**Electric Osmosis in Methylic Alcohol.**—A. Baudouin.—In methylic alcohol, as in water, the action of polyvalent ions is as follows:—A positive polyvalent ion (1) has little or no action on the charge of a positively charged substance in contact with the alcohol; (2) diminishes, and in certain cases reverses, the charge of a negatively charged substance. A negative polyvalent ion (1) has little or no influence on the charge of a negatively charged substance in contact; (2) diminishes, and in certain cases reverses, the charge on a positively charged substance.

**Preparation of Samaria and Atomic Weight of Samarium.**—G. Urbain and H. Lacombe.—(See p. 277).

**Formation of Hydrogen Silicide by Direct Synthesis from the Elements.**—Em. Vigouroux.—The author refers to a paper in *Comptes Rendus* of April 25, in which M. Violle gives an account of his preparation of hydrogen silicide direct from its elements. The author claims priority, and refers back to a paper of his own on July 18, 1901, in which he describes a preparation of hydrogen silicide direct from its elements.

**Apparent Volatilisation of Silicon in Hydrogen.**—A. Dufour.—In Geissler's tubes containing hydrogen arsenide the arsenic deposited by the discharge is displaced by simple distillation from the hot towards the cooler part of the tube whilst a current from the induction coil is traversing it. In tubes containing hydrogen silicide, the displacement of the silicon under the same conditions is explained by the formation of hydrogen silicide in the hot parts of the tube and its decomposition in Faraday's dark region. The volatilisation of the silicon is apparent only at this point.

**A Property of Tin-aluminium Alloys.**—Hector Pécheux.—When a little stick of either of the tin-aluminium alloys,  $\text{Sn}_2\text{Al}$ ,  $\text{Sn}_3\text{Al}$ ,  $\text{Sn}_4\text{Al}$ ,  $\text{SnAl}_3$ , whose surface has just been burnished, is plunged into cold water (at 13°) an abundant evolution of gas takes place round the polished part of the stick. The explanation of this is that, except at the tempered surface, the actual tin-aluminium alloy is not formed, but the metal consists of separate molecules of the two elements. Therefore a series of little thermoelectric elements exist of tin and aluminium, which decompose the water immediately. The tin and the aluminium, by reason of their very decided difference of specific heat ( $\text{Al } 0.218 \text{ cal.}$ ,  $\text{tin } 0.0562 \text{ cal.}$ ), are not at the same temperature (the molecules having been heated by the rubbing of the burnisher). An electromotive force is produced

which only ceases when the temperature of the two sets of molecules becomes equalised.

**Differentiation of Primary, Secondary, and Tertiary Alcohols of the Fatty Series.**—André Kling and Marcel Viard.—The authors have found a simple and rapid method of immediately concluding whether a given alcohol belongs to the primary, secondary, or tertiary group, by using the property of the unequal resistance of alcohols towards heat. Tertiary alcohols, if easily dehydrated with formation of non-saturated carbides, decompose into two molecules at the temperature of ebullition of naphthalene (218°), whilst the primary and secondary alcohols remain intact. If, however, the temperature is raised to 360°, the boiling-point of anthracene, the primary alcohols alone resist, both the secondary and tertiary ones being decomposed. If the vapour-density is taken by Meyer's method, first in the vapour of naphthalene, and then in the vapour of anthracene, it can at once be found, by the density determined, whether the alcohol is primary, secondary, or tertiary.

**Formation of Chloroanilines.**—Eyvind Bøedtker.—During the preparation of aniline the author sometimes observed that the product of reduction of nitrobenzene by tin and hydrochloric acid is not always aniline alone. This body is accompanied by others boiling at a temperature above 180°. In two cases there was no aniline at all in the product. On distilling these other products with steam, large crystals are deposited on cooling, as well as an oil. These crystals, when re-crystallised from alcohol, fuse at 70.5° and distil at 232.3°. Analysis proves the product to have the formula  $C_6H_4ClNH_2$ , which is chloraniline, and the author believes the reaction to take place according to the equation  $C_6H_5NO_2 + HCl + 2H_2 = C_6H_4\overset{Cl}{\underset{NH_2}{<}} + 2H_2O$ .

*Bulletin de la Société Chimique de Paris.*  
Series 3, Vol. xxix., No. 16.

**Law of Thermic Constants, and the Heat of Formation of Compounds of Barium.**—D. Tommasi.—Already inserted in full.

**New Method for the Detection and Estimation of the Smallest Traces of Arsenic.**—Armand Gautier.—Already inserted in full.

**Estimation of Arsenic in Sea-water, Sea-salt, Rock-salt, Mineral Waters, &c., and in some Ordinary Reagents.**—Armand Gautier.—Already inserted in full.

**Purification of Sulphuretted Hydrogen for the Detection of Arsenic.**—Armand Gautier.—Already inserted in full.

**Action of Persulphate of Ammonia on the Metallic Oxides.**—A. Seyewetz and P. Trawitz.—Already noticed.

**Oxidation of Ammonia and of the Amines by Catalytic Action.**—A. Trillat.—Already noticed.

**The Nitration of Acetylgaicol.**—F. Reverdin and P. Crepieux.—The authors wished to see whether the toluene-*p*-sulphonic ether of gaicol would give on nitration a product having a different constitution to that given by acetylgaicol, and they repeated Barbier's experiment to form a nitroacetylgaicol fusible at 135–136°. By operating in the cold they obtained, not a nitroacetylated derivative, but a yellow body fusing at 122°, corresponding to the dinitrogaicol of which the acetylated derivative fuses at 115°. By operating on the water-bath, they obtained the mononitroacetylgaicol of Meldola, fusible at 101°. They repeated this nitration with various proportions of acetic and nitric acids, and with nitric acid of different density, and in every case they obtained the mononitroacetylated derivative fusible at 101°. An interesting conclusion is to be drawn from these experiments, viz., that the nitration of acetylgaicol under the given conditions, and in the cold, gives rise to a de-acetylation and the formation of the dinitro-derivative, while, by

operating with heat, the acetyl resists, and the mononitroacetylated derivative is formed.

**Some Derivatives and Products of Condensation of  $\beta$ -Oxy- $\alpha$ -naphthoic Aldehyde.**—A. Helbronner.—The author has continued his examination of  $\beta$ -oxy- $\alpha$ -naphthoic aldehyde, and has prepared the following derivatives:—Acetate of  $\beta$ -oxy- $\alpha$ -naphthoic aldehyde and benzoic ether; also other bodies, by its condensation with cyanacetate of ethyl and with acetylacetone.

**Oxide of Ethylene from the  $\beta$ -Cyclohexanediol-1, 2 and its Derivatives.**—Leon Brunel.—Already noticed.

**Action of Ammonia on the Oxide of Ethylene of the  $\beta$ - $\alpha$ -Cyclohexanediol.**—Leon Brunel.—Already noticed.

**Stachyose.**—C. Tanret.—This is a long paper in which the author describes the preparation, constitution, and physical properties of stachyose, such as its solubility, rotatory power, and crystalline form. With the exception of their crystalline form, the author finds no difference between stachyose and mannetotetrose. On the other hand, M. Wyruboff finds that their crystalline forms are identical, and that they are one and the same sugar, namely, a tetrose with the formula  $C_{24}H_{42}O_{21}$ .

**Estimation of Essence of Turpentine from Landes.**

—M. Vèzes.—The author set himself a double problem—that of estimating the normal and the abnormal adulterants of essence of turpentine. The normal adulterants are oil of resin and colophane. The abnormal ones practically possible can be defined by two conditions—(1) They must be mixed with the essence without sensibly modifying its appearance; (2) their price must be lower than that of the essence. These two conditions are only fulfilled by a limited number of substances, namely, oil of petrole, white spirit or oil of schist, essence of petrole-ether, benzine, and sulphide of carbon. Methods for the detection and estimation of all these substances are given.

**An Oxidising Bacterium: its Action on Alcohol and Glycerin.**—R. Sazerac.—While examining a wine vinegar the author obtained cultures which, after a short time, reduced the cupropotassic reagent. The microbial growth contained a microbe very different in form and in the appearance of its cultures from the *Mycoderma aceti* and the bacterium of sorbose. When carefully isolated on plates of glycerinated gelose at 2 per cent, it constantly gave, with glycerin broth, homogeneous cultures possessing this reducing power at ordinary temperatures. This microbe is fairly large, and is easily stained with the basic aniline colours, but not by Gram. It will not grow in meat broth, nor on potato, but grows well on glycerin gelose. The author has proved that it is capable of rapidly oxidising glycerin, transforming it into dioxyacetone.

**New Electric Accumulator.**—D. Tomassi.—The novelty in this accumulator is in the form of the lead plates, which are designed to effect the circulation of the electrolyte and its diffusion through the active matter. An efficiency is claimed of 22 to 24 ampère-hours per kilogram. of plates.

## MISCELLANEOUS.

**Bromides of Sulphur.**—O. Ruff and G. Winterfeld.—With the object of settling the question of the existence of bromides of sulphur containing more bromine than  $S_2Br_2$ , the authors, after having prepared this latter in a state of purity by heating the elements in a sealed tube to 100° and rectifying the product under reduced pressure, then mixed it with known quantities of pure bromine, and measured the fusion-points as well as the vapour tension of a large number of such mixtures. They have not succeeded in preparing double bromides related to  $SBr_4$ , as takes place with  $SCl_4$ . They come to the conclusion that bromides of sulphur, other than  $S_2Br_2$ , do not exist.—*Berichte*, vol. xxxvi., p. 2437.

**Iodometric Estimation of Copper by means of Xanthate of Potassium.**—E. Rupp and L. Krauss.—Xanthate of potassium reacts on iodine by equal molecules, and the results of the iodometric estimation are the same both by direct addition and by titrating back the excess of iodine. *Preparation of the Reagent.*—Precipitate the solution of xanthate in absolute alcohol with anhydrous ether, or dissolve the commercial reagent in water, and clarify the solution by precipitation with a little sulphate of copper, and filter. The most suitable strength is 2.5 per cent. *Titration of the Reagent.*—Add to a known volume of the reagent some bicarbonate of soda and starch emulsion, then run in decinormal iodine solution until the blue colour persists for ten to twenty seconds. *Estimation.*—The cupric solution to be estimated is treated with acetate of soda if it is acid, then with a known volume of the xanthate, and 0.5 gm. of bicarbonate of soda. Make up to 100 c.c., filter, and estimate the excess of xanthate on 50 c.c. of the filtered liquid. Two molecules of xanthate correspond to two molecules of iodine and to one atom of copper. The results are accurate to about 0.5 per cent, more or less.—*Berichte*, vol. xxxv., p. 4157.

**A Complex Double Salt of Manganous and Tungstic Acids.**—A. Just.—To a boiling solution of 100 grms. of  $\text{WO}_4\text{Na}_2$  in 100 c.c. of water we add 5 grms. of  $\text{SO}_4\text{Mn}$  dissolved in 10 c.c. of water; this gives a yellowish-white, flocculent precipitate of  $\text{WO}_4\text{Mn}$ . We then add 14 grms. of persulphate of ammonium, taking care to stir well. The solution soon becomes rose coloured, and then deep red; boil for a quarter of an hour, adding water to make up for that evaporated; then add an equal volume of water, and filter under pressure to remove a little  $\text{MnO}_2$  formed. On cooling slowly, the liquor deposits orange coloured crystals, which are drained and dried by pressing between filter-paper. The salt obtained is fairly soluble in warm water, but the solution formed soon decomposes, even in the cold, depositing  $\text{MnO}_2$ . Thus we cannot re-crystallise it in pure water, but only in the presence of an excess of  $\text{WO}_4\text{Na}_2$ . Its composition is  $3\text{Na}_2\text{O} \cdot 5\text{WO}_3 \cdot \text{MnO}_2 + 18\text{H}_2\text{O}$ , and the author gives the anhydrous salt the constitution  $\text{Mn}^{IV}(\text{WO}_4\text{Na})_4 \cdot \text{WO}_4\text{Na}_2$ . With the metallic salts it gives precipitates, even with the salts of potassium; the potassic precipitate is amorphous, but crystallises after some time.—*Berichte*, vol. xxxvi., p. 3619.

### NOTES AND QUERIES.

\* \* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

**Vacuum Machinery.**—Can any reader oblige us with the name of the makers of vacuum machinery for the extraction of moisture from jellies?—ELLIOTT & SONS.

### MEETINGS FOR THE WEEK.

WEDNESDAY, 15th.—Microscopical, 8. "A Direct Proof of Abbe's Theorems on the Microscopic Resolution of Gratings," by Prof. J. D. Everett. "Report on the Recent Foraminifera of the Malay Archipelago" (Part XVI.), by F. W. Millett. "Nature's Protection of Insect Life," by F. Enock.

Chemical, 5.30. "The Mechanical Analysis of Soils, and the Composition of the Fractions resulting therefrom" and "The Effect of Long-continued Use of Sodium Nitrate on the Constitution of the Soil," by A. D. Hall. "Decomposition of Oxalates by Heat" and "Some Alkyl-derivatives of Sulphur, Selenium, and Tellurium," by A. Scott. "Ultra-violet Absorption Spectra of certain Enol-keto-tautomerides—Part I., Acetyl-acetone and Ethyl Acetoacetate," by E. C. C. Baly and C. H. Desch. "Action of Acetyl Chloride on the Sodium Salt of Diacetyl-acetone, and the Constitution of Pyrone Compounds," by J. N. Collie. "Our present Knowledge of the Chemistry of Indigo," by W. P. Bloxam.

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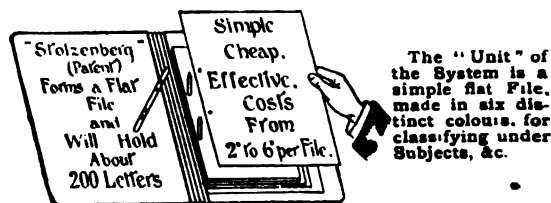
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### CONTENTS.

| ARTICLES:—                                                                                                                                                                                                                                                                                                                                                                                                                                        | PAGE |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| Radio-activity and Matter, by Clemens Winkler.....                                                                                                                                                                                                                                                                                                                                                                                                | 289  |
| Report on the Determination of Nitrogen, by F. W. Morse ....                                                                                                                                                                                                                                                                                                                                                                                      | 291  |
| Basic Chlorides of Copper, by André Brochet .....                                                                                                                                                                                                                                                                                                                                                                                                 | 293  |
| The Electrolysis of Alkaline and Alkaline-earth Chlorides with an Anode of Copper, by André Brochet .....                                                                                                                                                                                                                                                                                                                                         | 293  |
| PROCEEDINGS OF SOCIETIES—                                                                                                                                                                                                                                                                                                                                                                                                                         |      |
| CHEMICAL SOCIETY.—Imino-ethers and Allied Compounds corresponding with the Substituted Oxamic Esters—Action of Heat on $\alpha$ -Hydroxycarboxylic Acids, $\alpha$ -Hydroxystearic Acid—Ionisation and Chemical Combination—Ionisation and Chemical Combination in the Liquefied Halogen Hydrides and Hydrogen Sulphide—Some Compounds of Aluminium Chloride with Organic Substances containing Oxygen—Constituents of Chaulmoogra Seeds—&c. .... | 293  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES .....                                                                                                                                                                                                                                                                                                                                                                                                       | 297  |
| MISCELLANEOUS .....                                                                                                                                                                                                                                                                                                                                                                                                                               | 299  |

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ON

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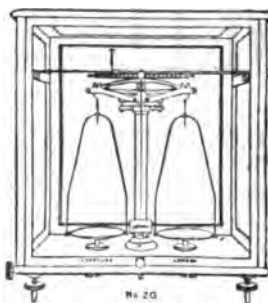


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# THE CHEMICAL NEWS.

VOL. LXXXIX., No. 2325.

## RADIO-ACTIVITY AND MATTER.

By CLEMENS WINKLER.

By the discovery of the phenomenon of radio-activity there has been opened up to physical, and presumably to chemical research also, a region which promises to surpass all our expectations in the peculiar and striking characteristics which it will reveal. We are confronted with a source of energy which seems to flow out continuously and spontaneously from itself, while we can only guess at its origin; with manifestations of energy which do not resemble those already known either in character or mode of expression; with substances which are like the elements in all particulars, and yet seem to possess a new nature. The most typical of these substances is radium, which in the opinion of its discoverer (*cf.* M<sup>me</sup>. S. Curie, "Untersuchungen über die Radio-activen Substanzen," Uebersetzt und mit Literatur, Ergänzungen Verschen, von W. Kaufmann, Braunschweig, 1904, S. 2) is to be regarded from a chemical point of view as a new element, and by its inclusion in the Atomic Weight Table of 1904 of the International Atomic Weight Committee is recognised as such.

Far less is definitely known about the other radio-active substances which have been discovered. The existence of polonium, discovered by P. and S. Curie (*Comptes Rendus*, vol. cxxvii., p. 175; *Chem. Centralbl.*, 1898, ii., 572) was for a long time regarded by many as doubtful, but it appears to have been revived in W. Marckwald's radio-tellurium (*Berichte*, 1902, xxxv., 2285 and 4239; 1903, xxxvi., 2662). Investigations which are certainly comprehensive and specially remarkable for the chemical work involved in them have been performed with radio-lead (K. A. Hofmann and E. Strauss, *Berichte*, 1900, xxxiii., 3126; 1901, xxxiv., 8, 907, 3033, 3970; K. A. Hofmann, A. Korn, and E. Strauss, *Berichte*, 1901, xxxiv., 407; K. A. Hofmann and V. Wölfl, *Berichte*, 1902, xxxv., 1453; 1903, xxxvi., 1040); but without taking into account F. Giesel's objections raised against them (*Berichte*, 1900, xxxiii., 3569; 1901, xxxiv., 3772) they have up to the present not been so far successful as to entitle us to regard it with absolute certainty as a new element. Again, the same might be said of A. Debiere's actinium (*Comptes Rendus*, vol. cxxix., p. 593; vol. cxxx., p. 906; *Chem. Centralbl.*, 1899, II., 1003; 1900, I., 1059), and of other radio-active substances such as have been supposed to have been discovered, for example, in the earths of the cerium and yttrium group (K. A. Hofmann and E. Strauss, *Berichte*, 1900, xxxiii., 3126; F. Giesel, *Berichte*, 1903, xxxvi., 342).

This uncertainty is quite explicable if we consider that for the performance of all researches up to the present at the most only very small quantities of pure or even enriched substance have been at the disposal of the investigators, for which reason the study of their properties, specially of those to be regarded as chemical, has been rendered much more difficult. Moreover, the study of the chemical behaviour of the substances in question naturally became of secondary importance, because of the great incentive to research which was given by the examination of the mysterious phenomenon of radio-activity with the use of the photographic plate, the electroscope, and phosphorescence screen, and if it has been by no means neglected it has certainly not received the attention that is usually paid to it when the characteristic nature of newly discovered elements is being established.

It has been found that the working up of the raw material containing radium may be performed by a method which is

very simple in itself, and in essentials follows the method of preparation of barium. In this method, by making use of certain differences of solubility, the method of fractionation has been successfully employed, and we have thus become acquainted with a characteristic radium spectrum. What importance, however, is to be ascribed to the spectral reaction is a question which, for the present, must remain undecided. In the case of a substance which has been separated from the very complex material of the Joachimsthal residues, and then only in exceedingly small quantities, it is quite possible that the spectral reaction might prove to be misleading. Apparently there exists little agreement between the spark spectrum of radium described by Demarçay (*cf.* S. Curie, *loc. cit.*, p. 20) and the flame spectrum described by F. Giesel (*Berichte*, 1902, xxxv., 3608), and the orange-red lines in the spectrum of radium bromide as well as the red colouration which this salt imparts to the flame may possibly be due to a foreign residue of the metals of the alkaline earths. Moreover, no compounds of radium have been sufficiently accurately examined, nor do we know of definite reactions peculiar to it. Strictly speaking, the only positive evidence in favour of its promotion to the position of a separate element is afforded by the determination of its atomic weight, which is so high as to justify the conclusion that radium, taking into account its similarity to barium, forms a new member of the metals of the group of the alkaline earths.

Radio-lead has been chemically more thoroughly examined than radium, without any considerable difference between its reactions and those of ordinary lead being detected. It was thought that in its atomic weight also a decided divergence had been obtained (K. A. Hofmann and E. Strauss, *Berichte*, 1901, xxxiv., 8, 907). The determination of the atomic weight of radio-lead was made by ascertaining the amount of sulphuric acid in the sulphate. The amount of SO<sub>4</sub> in the sulphate was fixed at 41.35 per cent, while that of lead sulphate only amounts to 37.71 per cent.

From this result, assuming that radio-lead, like thorium and uranium, is tetravalent, it was calculated that its atomic weight is 260.2, while that of lead is only 206.9. The correctness of the determination seems, however, doubtful because the sulphate with which it was carried out, after driving off the excess of sulphuric acid, was not heated until it began to glow, but was only dried at 400—420°. At such a low temperature experience has proved that it is not possible to expel completely the free acid which adheres to it, and thus it is not surprising to find the sulphuric acid of the sulphate examined higher than that of lead sulphate. The atomic weight of radio-lead cannot of course be deduced from it, and also the supposition, founded on the results of other sulphuric acid determinations which were affected by the same error, that possibly the homologue of manganese (the long sought ekamanganese of Mendeleeff) and of tin had been discovered, falls to the ground.

The researches, so admirable in all other respects, on the radio-activity of substances cannot, generally speaking, be regarded from a chemical point of view as adequate. Even the results of fractionation are to be accepted with circumspection, because they may easily give rise to fallacies. When the discovery of spectral analysis threw the world into a state of agitation similar to that now excited by the discovery of radio-activity, it was believed in all seriousness that calcium had been decomposed, because it had been found possible to cause the disappearance of the green line of its spectrum in one of the parts obtained by fractional precipitation of a solution of calcium chloride. and in connection with this crystallographers even began to make observations on the striking multiplicity of the calc spar form. Similar and certainly pardonable errors could occur in other cases, so that it can by no means be definitely stated that this has actually happened in the fractional treatment of radio-active substances.

It may now be asked whether it is generally established that the presence of a new element in a substance may be

presumed simply from the fact that it is radio-active. It would then be very remarkable if every one of the numerous elements, long known themselves, in which radio-activity has been observed, possessed a new attendant, also to be regarded as elementary, of practically perfect similarity of chemical behaviour. Many arguments may be brought forward against such an assumption quite apart from the fact that it is contrary to the present conception, which is not yet destroyed, regarding the nature of the elements, as well as to all experience. Uranium, an element undoubtedly to be regarded as typical in such a case, according to Sir William Crookes (*Proc. Roy. Soc.*, lvi., 409; *Chem. Centralbl.*, 1900, II., 364), may be decomposed into an active and an inactive part simply by treatment of its hydrous nitrate with ether, or by fractional crystallisation or fractional decomposition of the same compound by heating without either part showing the least difference from the other as regards its chemical behaviour. Béla v. Lengyel (*Berichte*, 1900, xxxiii., 1237), who expressed doubts as to the existence of radium, made barium active by heating its nitrate with uranyl nitrate, and Henri Becquerel (*Comptes Rendus*, cxxxi., 137; *Chem. Centralbl.*, 1900, II., 422), by precipitation with sulphuric acid of a solution of barium chloride to which active uranyl chloride had been added, prepared a strongly active barium sulphate, while the activity of the uranium experienced simultaneous diminution. Also K. A. Hofmann and E. Strauss (*Berichte*, 1900, xxxiii., 3126; 1902, xxxv., 1453) have obtained inactive fractions from active uranium compounds; on the other hand, they have succeeded in completely withdrawing the activity from uranium by means of barium or bismuth compounds. But the experiments of A. Debiere (*Comptes Rendus*, cxxxi., 333; *Chem. Centralbl.*, 1900, II., 557) are specially noteworthy; they were carried out by a similar method, but actinium was used, and by means of them it was shown that it was easily possible to make the compounds of barium active in such a way that they could scarcely be distinguished from those of radium, except for the fact that they gave no radium spectrum, and showed a decrease of activity after a time. The barium which has been made active is said to stand between barium and radium as regards its properties. Its chloride, like that of radium, is self-luminous when anhydrous, and also by fractional crystallisation may be decomposed into a more active and a less active part.

Only by induction can such striking observations be explained. Also the objection to Béla v. Lengyel raised by F. Giesel (*Berichte*, 1900, xxxiii., 1665), that the activity of the barium first caused by it may be produced by the radium contained in the uranium salt used, was quashed, because, as has been described, the same result has been obtained under other conditions, and also on a far large scale.

And what is to be said regarding the supposed material emanation of radio-active "elements," their instability, their decomposition with splitting off of helium, and the decomposition of the elements themselves? So far no proof has been furnished that elements of high atomic weight, among which gold and platinum are to be reckoned, are polymers of elements of low atomic weight, and that they could be decomposed into the latter. The conception of an element still retains its former meaning, and quite different and more thorough chemical experiments would be necessary to destroy it. But it appears every day more plainly that radio-activity seems to be extraordinarily widely diffused, and this idea leads to the question whether it may not be a purely physical process, a phenomenon which may adhere to matter without influencing its chemical composition something like the magnetism of magnetic iron ore, which, like it, may be increased, transferred, apparently annihilated, and again produced, and which also shows itself as a mysterious expression of energy proceeding from the substance, though nobody imagines that in magnetic ferroso-ferric oxide the presence of another element is to be assumed different from that in non-magnetic iron oxide. The idea of material difference will not occur to anybody,

since Fr. Heusler (*Schriften d. Gesellsch. z. Beförd. d. Gesammt. Naturwissenschaften z. Marburg*, 1904, xliii., 255; and *Zeit. für Angew. Chem.*, 1904, 260) succeeded in preparing alloys capable of magnetisation from non-magnetic metals such as manganese, tin, antimony, and aluminium. As regards radio-activity also we should be independent of the assumption of a material difference if the statement of F. Richarz and Rudolf Schenck (*Sitzungsber. d. Königl. Preuss. Akademie d. Wissenschaften*, 1903, lii.; 1904, ii.) remained without valid objection, viz., that oxygen on ozonisation becomes radio-active in such a way that it acts upon the photographic plate, causes intense luminescence of Sidot's blende (but not of other substances), and like radium gives rise to development of heat.

Considering the excitement about radium which now pervades the world, and makes itself felt to no small degree in lay circles, it is rather galling for chemists that they cannot say more about radium, which was discovered almost six years ago, than that it is exactly like barium except that it has a higher atomic weight, and possesses the wonderful spontaneous radiation. The chemical nature of radium is as yet almost unknown, and yet questions are often put about it, especially in those places in which pitchblende has been discovered, and where people are already dreaming of a most promising "radium mining" and a future "radium industry." So far as is yet known the occurrence of radio-active substances is entirely bound up with that of uranium. This is striking in the highest degree and cannot be explained if we are to assume the presence of special elements in these substances. For if the occurrence together of certain fundamental substances is by no means extraordinary and can be explained from their position in the system, their valence, the isomorphism of their compounds, and other similar characteristics, yet we never meet with the exclusive occurrence together of any substances, as would appear to be the case here. Thorium is, perhaps, an exception, or at any rate G. F. Barker (*Chem. Zeit.*, 1903, 522) ascribes to this element a radio-activity independent of the simultaneous occurrence of uranium. K. A. Hofmann and F. Zerban (*Berichte*, 1902, xxxv., 531; 1903, xxxvi., 3093) are of the contrary opinion, and F. Zerban (*Berichte*, 1903, xxxvi., 3911) also thinks that he has proved the presence of a small quantity of uranium in monazite sand, which has hitherto been supposed to be free from uranium. However, objection may be made to the method of research used by him, which originated with Laube (*Zeit. für Angew. Chem.*, 1889, 575), and was meant originally, not for analytical purposes, but for the working up of uranium residues.

The material for obtaining radium and other radio-active substances has hitherto been chiefly furnished by the residue obtained during the working of pitchblende for uranium preparations at the K. K. Österreich-Uranfabrik in Joachimsthal. This residue remains on solution in dilute sulphuric acid of pitchblende which has been previously roasted, ignited with soda and saltpetre, and washed with water. It consists essentially of gangue, silicic acid, iron oxide, basic iron sulphate, and lead sulphate, but also contains some bismuth and silver. Its weight amounts to 40 per cent of the weight of the uranium ore worked up, and it may be estimated that during the fifty years the Joachimsthal Uranfabrik has existed 150—200 tons of this residue have been made. The total amount of radio-active substances contained in it is difficult to estimate, but it would amount to a few grms. only of radium and to fractions of a gm. of radio-tellurium.

The residue in question possesses 4 to 5 times the activity of metallic uranium. 1000 kilograms of it, according to S. Curie, yielded 10—20 kilograms of raw sulphate of 30—60 times the activity of uranium, and this again gave 8 kilograms of barium chloride containing radium with activity 60 times that of uranium. From this it seems to follow that the increase of activity does not keep pace with the enriching of the barium compounds, but falls far short of it. Thus it does not seem to form a trustworthy

standard for the increase of the amount of radium present. Even a chloride brought by fractional crystallisation to 3500 times the activity of uranium must have consisted essentially of barium chloride, as the atomic weight of the metal contained in it amounted only to 140 and was thus not much higher than that of barium. No information is at hand concerning the activity of the presumably purest radium chloride, which still, however, contains some barium, but the best radium preparations are said to be 50,000 to 100,000 times more active than uranium (O. Dammer, "Handb. d. Anorg. Chemie," vol. iv., p. 952).

A question of great importance is: In what form of combination is the radium contained in pitchblende and in the residue resulting on its solution? In the latter case most decidedly as sulphate, and probably also in the former, because heavy spar ordinarily accompanies pitchblende. As now the solubility of the sulphates of the metals of the alkaline earths decreases with rise of atomic weight, radium, as the last member of the group, must form the least soluble sulphate. Hence, and from the isomorphism of the barium and radium compounds (F. Rinne, *Centralbl. f. Min. u. Geol.*, 1903, 134), it must be concluded that the heavy spar accompanying pitchblende is the real carrier of the radium contained in the Joachimsthal ore. But Sir William Crookes (*Proc. Roy. Soc.*, lvi., 409; *Chem. Centralbl.*, 1900, II., 364) has not succeeded in proving the existence of radio-activity in any of the heavy spar specimens examined by him, and, even if amongst them that from Joachimsthal was not included, this is strange, inasmuch as according to all experience relating to the occurrence together and mutual substitution of the elements, it is not to be assumed that radium, unlike calcium and strontium, is excluded from appearance in natural barium compounds, if it stands beside barium actually as an independent element. But if heavy spar containing radium exists, the radium it contains will pass over into the barium chloride prepared artificially from it, and then even if it were ever so small in quantity it could not be difficult to concentrate it by fractional crystallisation of this salt on a large scale. The experiment with 50 kilograms of commercial barium chloride planned for this purpose by S. Curie has only given negative results.

But even if radium for some unknown reason can only occur with uranium it must, at any rate in the Erzgebirg, be found comparatively widely distributed. For besides the actual places in which pitchblende has been found, like Joachimsthal or those at present more or less worked out, e.g., Johanngeorgenstadt, Schneeberg, Annaberg, and Freiberg, we find in the region named above rock containing very little uranium, particularly granite, in which the presence of uranium may be shown by a small quantity of uranite and uranocircite, which acts on the photographic plate, and sometimes also is visible in the paving material of roads. Hence it seems that it may not be impossible to discover, in that region in which the occurrence of bismuth is very widely extended, raw materials which may be successfully worked for radio-active substances. By the preparation of these latter pure in larger quantities and their thorough chemical examination we shall perhaps succeed not only in approaching some explanation of the nature of radio-activity, but also in establishing without any doubt the existence of radio-active elements of special nature and definite chemical behaviour.

**Use of Berthelot's Calorimetric Bomb to prove the Existence of Arsenic in the Organism.**—Gabriel Bertrand.—M. Berthelot has already used his calorimetric bomb for the estimation of various simple bodies present in organic matters. The author has applied this method, with great success, to the detection of traces of arsenic. In this paper he describes the details of the method he has employed, as well as other experiments on the limit of sensibility, control experiments, &c. He finds that arsenic is present normally in the shell of the turtle, in sponge, the white of egg, the thyroid gland, and other substances.—*Bull. Soc. Chim.*, Series 3, xxix., Nos. 18—19.

## REPORT ON THE DETERMINATION OF NITROGEN.\*

By F. W. MORSE.

(Concluded from p. 284).

### Comments of Analysts.

**Department of Foods and Feeding, Hatch Experiment Station.**—Mechanical conditions were not uniform; a considerable portion of each sample was coarse. The substances should be reduced so as to pass a sieve of No. 2 perforated sheet metal (400 perforations to the square inch). This is especially true of mixtures containing highly nitrogenous foods; otherwise sampling for nitrogen determinations or availability, where only a small amount is employed, is very unsatisfactory, and concordant results are practically impossible except by chance.

In the determination of total nitrogen, by the plain Kjeldahl method, the reagents used in the digestion were one-half gm. re-sublimated metallic mercury (B. and A.), 25 c.c. chemically pure sulphuric acid, specific gravity 1.84 (B. and A.); and, in the distillation, 200 c.c. distilled water, 25 c.c. potassium sulphide solution, 1—25 (Merck), 100 c.c. sodium hydrate solution, 1—2 ("Red heart"), one-half gm. granulated zinc (30 mesh, B. and A.).

In the determination of total nitrogen by the plain Kjeldahl method plus sulphate, the reagents used were the same as in the plain Kjeldahl with the addition of 10—12 grms. of crystallised potassium sulphate (B. and A.), to facilitate the digestion and to insure perfect oxidation.

The results were as follows:—

1. More perfect oxidation; this is noticeable in every case where cotton-seed meal is present; the average increase in nitrogen for the samples is 0.056 per cent, 3.943 per cent, and 3.887 per cent.

2. More rapid oxidation; the solutions cleared—i.e., bulk of organic matter was destroyed in twenty-one minutes (average) as against forty-one minutes in the plain Kjeldahl method.

3. There is less reaction upon the addition of water in preparing for the distillation than in the case of the plain Kjeldahl method.

The mechanical condition prevented the obtaining of closely agreeing results in both methods.

The neutral permanganate solution was prepared from pure crystallised reagent (Merck). The plain Kjeldahl method plus sulphate was used for the determination of the nitrogen in the insoluble residue. Unevenness of the samples and variation in action of the solvents probably account for the poor parallels. The objectionable features in the manipulation of this method are chances of error from repeated transferring, &c., length of time required, and difficult filtration.

The alkaline permanganate solution was prepared from pure crystallised potassium permanganate (Merck) and stick sodium hydrate, purified by alcohol (Merck). The results are apparently low. This can be accounted for, partly at least, by errors in the method as sent out:—

1. A weak, incorrectly prepared solution. Sixteen grms. of powdered crystallised potassium permanganate and 150 grms. of sodium hydrate should be dissolved in water, and after cooling made up to 1000 c.c.

2. Substances too coarse to receive the maximum effect of the solution.

3. Digestion too short. The heating should be maintained for one hour without distilling over, but not necessarily below ebullition, and the flask should be shaken occasionally to wash down adhering particles. The distillation should be conducted as usual, simply using care not to break the flask. The objectionable feature in the manipulation is the adhering of the material to the sides of the flask and the smallness of the solution with which to wash it down. Little or no frothing occurs after proper

\* Proceedings of the Nineteenth Annual Convention of the Association of Official Agricultural Chemists, held at Washington, D.C.

digestion (see the "Fourteenth Report of the Vermont Experiment Station," p. 219).

*C. H. Jones, Vermont.*—The filtrate after the digestion with neutral permanganate solution was colourless in each case.

With the alkaline permanganate method I am satisfied that the preliminary digestion of thirty minutes or one hour is necessary, not so much that the results may be slightly increased, but that danger from frothing is lessened if not entirely obviated when the distillation begins. It should be noted that it is quite necessary to rotate the flask occasionally during the digestion and distillation if the material shows a tendency to creep up the sides of the flask.

I re-ground Samples 1, 3, and 4 before making any tests, as the samples containing blood seemed to me too coarse to enable one to get concordant results with the Kjeldahl method.

*Y. B. Robb, Maryland.*—The total nitrogen was determined by the Gunning method, and the samples were digested for one hour.

In the alkaline permanganate method the flasks were shaken several times during the process of distillation to prevent the material adhering to the sides of the flask.

In my opinion the neutral permanganate method is very unsatisfactory, as the results obtained by it are not at all concordant.

*F. W. Woll, Wisconsin.*—No. 597, S. and S. filters 9 c.m. were used.

Sample No. 1, blood, washed clear.

Sample No. 2, cotton-seed meal, washed turbid, which necessitated re-filtering through double paper (No. 597, S. and S.).

Sample No. 3, washed clear.

Sample No. 4, washed turbid, treated the same as sample No. 2.

Jena 800 c.c. round-bottom flasks were used for distillation.

*A. M. Peter, Kentucky.*—It being apparent that all the permanganate was reduced before the digestion was completed in the alkaline method, an additional quantity of 1.6 grms. permanganate dissolved in 100 c.c. water were added to each of the flasks in set 3, and the distillation repeated, the following additional quantities of nitrogen being obtained:—Sample No. 1, 1.25 per cent; No. 2, 1.30 per cent; No. 3, 1.54 per cent; No. 4, 1.40 per cent. At the close of the distillation the liquid in the flasks was still green from excess of permanganate.

Another portion of 1.6 grm. permanganate and 100 c.c. water was added to each flask, and the distillation was repeated with the following results:—Additional nitrogen obtained from sample No. 1, 0.40 per cent; No. 2, 0.20 per cent; No. 3, 0.20 per cent; No. 4, 0.13 per cent.

The total available nitrogen obtained in these three distillations was as follows:—Sample No. 1, 3.22 per cent; No. 2, 2.76 per cent; No. 3, 3.08 per cent; No. 4, 3.01 per cent.

Another set of determinations was made, following the referee's directions in every respect, except that 200 c.c. of alkaline permanganate were used instead of 100 c.c. The results were that the following percentages of available nitrogen were obtained:—Sample No. 1, 2.24; No. 2, 1.90; No. 3, 2.17; No. 4, 2.10.

Still another series was made, following the referee's directions in every particular, except that the distillation was pushed until the residues in the flasks were dry at the bottom. In this set some parts of the residue may have been overheated, as the flasks were over the direct flame. The results were the following percentages of available nitrogen:—Sample No. 1, 2.58; No. 2, 2.16; No. 3, 2.93; No. 4, 1.50.

It appears that there is not enough permanganate in 100 c.c. of the alkaline solution as prescribed to complete the decomposition. I do not see that either method is giving us available nitrogen, and the alkaline permanganate, as carried out, seems to be especially faulty.

### Discussion of Results.

Without discarding any figures on total nitrogen, the differences between maximum and minimum results are, respectively, 0.60 in No. 1, 0.46 in No. 2, 0.36 in No. 3, and 0.40 in No. 4. These discrepancies are large for total nitrogen.

The point mentioned by the referee last year with regard to the time of digestion is brought out here, namely, that the digestion should be prolonged in order to insure the recovery of all the nitrogen.

Setting aside the highest and lowest results, we have determinations ranging within less than 0.3 per cent, which is as exact a result as has been obtained in the past.

When the results on the availability of the nitrogen are considered, it is seen that duplicate analyses by any one analyst agree well, taking each method by itself.

Both methods have been evolved from the method of determining albumenoid ammonia in water, as is shown by the first method proposed in the reports of the Association (U.S. Department of Agriculture, Division of Chemistry, *Bulletin*, 47). They differ radically in principle, the neutral permanganate being used merely to dissolve the nitrogen, while the alkaline method dissolves it, and also transforms it into ammonia.

The critical features of the former method are time and temperature, as in determining citrate soluble phosphoric acid. The latter method possesses the same weakness possessed by Wanklyn's method for albumenoid ammonia—that in the case of vegetable matter it will recover only about one-half of the total nitrogen, and its critical features are the amount of permanganate which will be affected by cellulose and the amount of distillate which is collected.

As is mentioned by Mr. Peter, neither method seems to give the available nitrogen; yet I can think of nothing better. We have in fertilisers about all forms of vegetable and animal proteids, both raw and cooked. Obviously none of the usual solvents for proteids can be employed in this work. We must therefore attempt to use a substitute.

The neutral permanganate method has compared most favourably with vegetation tests in its relative effects on the various nitrogenous fertilisers. On the other hand, the alkaline method is a convenient one for determining whether a fertiliser is easily transformed to ammonia or not.

This method needs to be modified somewhat. If more time had been available, the referee would have tried increasing the amount of solution used in order to have a greater volume for distillation and the regulation of the amount of distillate collected. Reasoning from the practice with albumenoid ammonia in water analysis, this latter point would be especially important. Another fact which bears on this point is that in residues after distillation, when washed as in the neutral method and the insoluble nitrogen determined, the nitrogen was found to have been almost totally dissolved, even in a sample of ground leather.

With regard to the neutral method I think that the use of a Witt plate or Hirsh funnel is, as a rule, advantageous in reducing the time and enabling one to use a smaller filter-paper.

I do not think it necessary to make a change in the method of determining total nitrogen, although the weight of evidence is in favour of prolonging digestion for three or more hours. The increase in nitrogen is so slight that it would be negligible in some work, while important in others. The good sense of our station chemists will result in their familiarising themselves with the work of the Association on such a point, after which the character of the work will govern the mode of execution.

I believe that the alkaline permanganate method is worthy of further attention, and that each method should be reduced to a form which will give more closely concordant results.

My only recommendation as a result of this year's work is that the subject be continued by the next referee, with the view of getting more concordant results with each method.

# BASIC CHLORIDE OF COPPER.

By ANDRÉ BROCHET.

THE crystallised basic copper chloride,  $\text{Cu}(\text{ClO}_3)_{2.3}\text{Cu}(\text{OH})_2$ , has been obtained by M. Bourgeois (*Bull. Soc. Chim.*, [3], vol. xix., p. 948) by decomposing chlorate of copper by heat. During my recent researches I have observed that this basic salt is obtained in the amorphous state under the same conditions as the basic chloride, either by the action of an alkaline or an alkaline-earthly base on the chlorate of copper in solution, or by the action of the hydrate of copper on an excess of chlorate in solution. All these reactions take place as well in the cold as when heated.

The dried product forms a greenish-blue powder analogous to the corresponding basic salts. It answers to the formula given by M. Bourgeois; however, it always contains an excess of hydrate of copper and a certain quantity of water, which cannot be got rid of, either by heating to  $100^\circ$  or *in vacuo*.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 6.

## THE ELECTROLYSIS OF ALKALINE AND ALKALINE-EARTHY CHLORATES WITH AN ANODE OF COPPER.

By ANDRÉ BROCHET.

MESSRS. Bancroft and Burrows have described the curious phenomena which take place when chlorate of potassium is electrolysed with an anode of copper. In a previous paper on this subject, I have given, for my own part, some explanation of these occurrences. I have extended this research to the more soluble chlorates of sodium and barium; barium has the further advantage of being easily detected and estimated in the precipitate.

Knowing the action of chloric acid, and of chlorate of copper on this metal, it is easy to complete this explanation. I have already stated that the anion,  $\text{ClO}_3$ , is decomposed on contact with the copper anode, and thus permits the attack of a greater quantity of the metal. This is due to a purely chemical action apart from the electrolytic one.

Now, in the case of the alkaline and alkaline-earthly chlorates and of chlorate of copper, this solution cannot be more than double the amount of copper precipitated on the cathode of the voltameter.

The manner in which the electrolysis of chloric acid and of chlorate of copper goes on, has led me to modify my first hypothesis and to admit that the decomposition of the ion,  $\text{ClO}_3$ , or of the chlorate of copper on contact with the anode, instead of being the principal cause of the attack of this anode, was the consequence of it.

The reaction which takes place is due simply to the fact that the copper goes into solution in the form of cuprous ions, but, as in certain electrolytes, cyanide of potassium, for example, the metal simply dissolves in the form of cuprous ions, and remains as such, in the present case the copper is dissolved in the form of cupric ions; further, the cuprous salts formed by the primary action, the chloride or the oxide, are more or less rapidly oxidised.

According to the conditions of temperature, density of current, concentration, &c., the amount of cupric salt formed primarily is more or less considerable, which explains why the ratio of the copper dissolved at the anode to the copper precipitated on the cathode of the voltameter is variable and hardly equal to 2.

It is not necessary here to give in detail the experiments made under the same conditions as those of Burrows, and which have given me analogous results. In a general manner, whether we have to do with the acid, with chlorate of copper, or with an alkaline or an alkaline-earthly chlorate, the reaction at the anode is the same.

In the case of the acid, the products formed pass into solution; with the salt of copper, they are found in the

form of double salts; with the alkaline or alkaline-earthly chlorates, the basic salts are partially decomposed by the alkali formed at the cathode, but the decomposition is not complete, and there remains in the precipitate of the chloride and of the chlorate a state of equilibrium with the alkali remaining in solution. The hydrogen at the cathode thus goes first to the chlorate, so that the precipitate will not contain any, then to the oxide to form metallic copper insoluble in dilute sulphuric acid, and soluble in hot concentrated acid with the evolution of sulphurous acid gas. For the above mentioned reasons the amount of copper contained in the precipitate will be less than that obtained in an analogous precipitate from a salt unable itself to furnish any element reducible by hydrogen, such as sulphate of sodium, for example.

Thus the black precipitate is very impure; in fact, it contains, besides the oxide of copper, metallic copper, chlorides, and the alkali corresponding to the salt electrolysed, as is easy to prove by using chlorate of barium.—*Bull. Soc. Chim.*, Series 3, vol. xxxi., No. 6.

## PROCEEDINGS OF SOCIETIES.

### CHEMICAL SOCIETY.

Ordinary Meeting, Thursday, June 2nd, 1904.

Dr. W. H. PERKIN, F.R.S., Vice-President, in the Chair.

MESSRS. T. L. D. Porter and C. Smith were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. William Herbert Dalton, 85, Hayter Road, Brixton Hill, S.W.; John Evans, 67, Surrey Street, Sheffield; Archie Willoughby Henzell, Witton Hall Industrial School, Birmingham; Arnold Bertram Tonkin, Durban, Natal; Henry Bridges Weeks, The Retreat, Infield Park, Barrow-in-Furness.

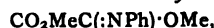
Of the following papers, those marked \* were read.

\*95. "Imino-ethers and Allied Compounds corresponding with the Substituted Oxamic Esters." By GEORGE DRUCE LANDER.

When piperidine and phenylhydrazine condense in anhydrous media with methyl dichloro-oxalate, the dipiperidide,  $\text{CO}_2\text{Me} \cdot \text{C}(\text{C}_5\text{NH}_{10})_2 \cdot \text{OMe}$ , and the imino-compound,  $\text{CO}_2\text{Me} \cdot \text{C}(\text{OMe}) \cdot \text{N} \cdot \text{NHPh}$  (m. p.  $123-124^\circ$ ), result, as described by Anschütz and Stiepel (*Annalen*, 1899, cccvi., 11). The latter substance gives the corresponding acid, which is unstable, yielding, on boiling with aqueous methyl alcohol, carbon dioxide, oxalic acid, and formazan.

With aniline and *p*-toluidine it was not found possible to isolate either the mixed ortho-compounds,  $\text{CO}_2\text{Me} \cdot \text{C}(\text{NHR})_2 \cdot \text{OMe}$ , or the imino-ethers,  $\text{CO}_2\text{Me} \cdot \text{C}(\text{NR}) \cdot \text{OMe}$  of Anschütz and Stiepel. Diaryl-amidino-oxalic esters,  $\text{CO}_2\text{Me} \cdot \text{C}(\text{NR}) \cdot \text{NHR}$ , and small quantities of the corresponding amidinoamides were obtained in the cold, and also in boiling xylene solution, together with substituted oxamates and oxamides, substituted formamides, carbon dioxide, and methyl chloride.

Semi-N-phenylimino-oxalic dimethyl ether—



a liquid (b. p.  $130-132^\circ/12$  m.m.) prepared by methylating methyl oxanilate by means of silver oxide, resembles the diethyl analogue (*Trans.*, 1901, lxxix., 699). Dry ammonia was found to be without action in the cold on any of the oxalimino-ethers.

The following compounds were described:—Methyl phenylmethyloxamate (b. p.  $170-175^\circ/13$  m.m.); semi-N-tolylimino-oxalic diethyl ether (b. p.  $160/14$  m.m.); methyl-p-diphenylamidino-oxalate (m. p.  $65-66^\circ$ ), and the analogous di-p-tolyl methyl ester (m. p.  $103^\circ$ ), and ethyl ester (m. p.  $98-100^\circ$ ).

The diphenyl- and di-*p*-tolyl-amido-oxalic acids lose carbon dioxide on heating, and change into substituted formamides,—



whilst the di-*p*-tolylamidino-ester, on heating, evolves carbon dioxide and yields di-*p*-tolylmethylformamidine. The hydrochlorides of the amidino-esters decompose on fusion, giving rise to methyl chloride, carbon dioxide, and formamides.

Methyl iodide and the di-*p*-tolylamidino-ester give the methyl ester,  $\text{CO}_2\text{Me}\cdot\text{C}(\text{NC}_7\text{H}_7)\cdot\text{NMe}\cdot\text{C}_7\text{H}_7$  (m. p.  $91-92^\circ$ ), the corresponding acid decomposing at  $80-81^\circ$  into carbon dioxide and the foregoing di-*p*-tolylmethylformamidine.

\*96. "The Action of Heat on  $\alpha$ -Hydroxycarboxylic Acids. Part I.  $\alpha$ -Hydroxystearic Acid." By HENRI RONDEL LE SUEUR.

The investigation of the action of heat on  $\alpha$ -hydroxystearic acid, briefly described in a preliminary note (*Proc.*, 1904, xix., 14), has now been completed, and in view of the fact that Blaise (*Comptes Rendus*, 1904, cxxxviii., 697) has recently published an account of work of a similar nature, it appears desirable to set forth the results of this investigation.

Margaric aldehyde,  $\text{C}_{16}\text{H}_{33}\cdot\text{CHO}$ , produced on heating  $\alpha$ -hydroxystearic acid, crystallises in needles, and melts at  $36^\circ$ ; its oxime and semicarbazone melt at  $89.5$  and  $107-108^\circ$  respectively. With hydrogen cyanide, it forms  $\alpha$ -hydroxyheptadecyl cyanide (m. p.  $61.5-62.5^\circ$ ), which, on partial hydrolysis, gives  $\alpha$ -hydroxystearamide melting at  $148-149^\circ$ , and, on complete hydrolysis,  $\alpha$ -hydroxystearic acid.

Margaric acid, obtained by oxidation of the above aldehyde with potassium permanganate, melts at  $60-61^\circ$ ; its methyl and ethyl esters melt at  $29^\circ$  and  $28^\circ$  respectively.  $\alpha$ -Bromomargaric acid crystallises from dilute acetic acid in glistening plates (m. p.  $52.5^\circ$ ).

#### DISCUSSION.

Dr. LEWKOWITSCH pointed out with regard to the  $\alpha$ - and  $\beta$ -hydroxystearic acids obtained from oleic and isooleic acids respectively that the  $\alpha$ -compound was not identical with the  $\alpha$ -acid described by the author, since, on oxidation with chromic acid, it yielded sebacic and ketostearic acids, and was probably a  $\kappa$ -acid. It would be of interest to know whether the author's margaric acid was identical with Kraft's margaric acid or with daturic acid; the latter might now be regarded as an acid which could be somewhat easily obtained, since it had been found in lard as the mixed glyceride, daturodistearin.

Dr. LE SUEUR, in reply, said that the acid which he had described was probably identical with Kraft's margaric acid, as the two substances melted at practically the same temperature; but, as Kraft had not prepared any characteristic derivatives from this acid, he could not state definitely that they were one and the same substance. These remarks also applied to daturic acid, which melts at  $55^\circ$ .

\*97. "Ionisation and Chemical Combination." By JAMES WALLACE WALKER.

The assumption that all chemical reaction takes place between pre-existing ions is unsatisfactory in that it is incapable of experimental demonstration. In many reactions, the formation of intermediate compounds points to the conclusion that such changes take place in several stages. Some of these compounds can only exist in virtue of the higher valencies possessed by their constituent atoms. Metathetic reactions between the alkyl halides, which take place with great readiness in presence of aluminium chloride, are described and shown to be due to the formation of such compounds; they are accompanied by the production of ionised solutions, but in an analogous case, where the solution is also ionised, the corresponding reaction is excessively slow. Several series of observations point to the conclusion that ionisation is the result of

chemical combination due to potential valency, and that reaction is not dependent in these cases on antecedent ionisation.

The ethers and the alkyl halides are shown to become quite good ionising media when employed in certain reactions in which they are themselves involved, although from their very low dielectric power they would not be expected to have this property. The variation of the molecular conductivity of some of these solutions with varying concentration shows very pronounced maxima and minima, which point to the formation of compounds with the solvent. These maxima and minima correspond in some instances with pronounced colour changes in the solutions. The conclusion is that, in general, combination through the operation of higher valencies precedes ionisation or any other manifestation of chemical change.

\*98. "Ionisation and Chemical Combination in the Liquefied Halogen Hydrides and Hydrogen Sulphide." By JAMES WALLACE WALKER, DOUGLAS MCINTOSH, and EBENEZER HENRY ARCHIBALD.

A large number of organic substances are shown to yield solutions which are good conductors of an electric current when dissolved in the liquefied halogen hydrides. Nearly all of those containing oxygen form conducting solutions in one or other of these media. In most of these instances, the ionisation can only take place after combination with the solvent. The importance of the existence of such compounds in depicting the mechanism of some of the fundamental reactions of organic chemistry is pointed out in the preceding communication. Hydrogen sulphide, although an exceedingly good solvent for nearly all classes of substances, yields only a few solutions which conduct at all and very few that conduct well. Almost all of these are, however, cases in which chemical combination between solvent and solute may be assumed.

\*99. "Some Compounds of Aluminium Chloride with Organic Substances containing Oxygen." By JAMES WALLACE WALKER and ARTHUR SPENCER.

Compounds of aluminium chloride with ether, anisole, ethyl benzoate, methyl mandelate, ethyl oxalate, ethyl malonate, acetic acid, *o*-nitrotoluene, and *m*-dinitrobenzene have been prepared in order to determine whether there is a proportionality between the number of atoms of oxygen and the number of molecules of aluminium chloride which enter into combination.

In determining the depression of freezing-point of aluminium chloride in *o*-nitrotoluene solution, it was found that this solvent has two crystalline forms, one melting at  $-10^\circ$  and the other at  $-4.25^\circ$ . The molecular weight of the salt apparently increases with increasing volume.

\*100. "The Constituents of Chaulmoogra Seeds." By FREDERICK BELDING POWER and FRANK HOWORTH GORNALL.

The seeds, which afford the chaulmoogra oil of commerce are derived from *Taraktogenos Kursii* (King), a native of Burmah, and not, as has until quite recently been assumed, from *Gynocardia odorata* (R.Br.). The oil has previously been examined by Moes ("Year-book of Pharmacy," 1879, 523-533), Petit (*Journ. Pharm. Chem.*, 1892, xxvi., 445), and more recently by Schindelmeyer (*Ber. Deutsch. Pharm. Ges.*, 1904, xiv., 164), but their results differ in many respects from those obtained by the present authors, which are as follows:—

The seeds of *Taraktogenos Kursii* (King) contain a hydrolytic enzyme, and also an unstable cyanogen compound, which reacts with the enzyme when the seeds are crushed, giving rise to hydrogen cyanide. Numerous attempts were made to isolate this compound, but without success. Further experiments will be made in this direction.

On expression, the seeds yielded 30.9 per cent of a fatty oil, which had the following constants:—M. p.  $22-23^\circ$ ; sp. gr. 0.951 at  $25^\circ$  and 0.940 at  $45^\circ$ ;  $[\alpha]_D^{15} +52^\circ$ ; acid value 23.9; saponification value 213; iodine value 103.2.



On hydrolysis, the fatty oil yielded glycerol, a very small amount of phytosterol,  $C_{26}H_{43}OH$  (m. p.  $132^{\circ}$ ), and a mixture of fatty acids (m. p.  $44-45^{\circ}$ ;  $[\alpha]_D +52.6^{\circ}$  in chloroform; acid value 215; iodine value 103.2), which consisted chiefly of several homologous acids belonging to a series  $C_nH_{2n-4}O_2$  containing a closed ring and one ethylenic linking, no member of which has hitherto been isolated from a fatty oil. The highest of these homologues present, which was isolated in a pure condition, separates from most of the usual organic solvents in glistening leaflets (m. p.  $68^{\circ}$ ; b. p.  $247-248^{\circ}/20$  m.m.,  $[\alpha]_D +56^{\circ}$ ), has the formula  $C_{18}H_{32}O_2$ , and is designated *chaulmoogric acid*. It combines with only two atomic proportions of bromine or iodine. Palmitic acid also was identified, and there is reason for assuming the presence of a near homologue or homologues of chaulmoogric acid, but belonging to the series having the general formula  $C_nH_{2n-4}O_2$  with two ethylenic linkings. Undecylic acid and hydroxy-acids were proved to be absent, and an individual acid corresponding with hypogaeic acid could not be isolated. The "gynocardic acid" of all previous investigators is believed to be a mixture of several substances.

The "press-cake" yielded, besides formic and acetic acids and a very small amount of volatile esters having the characteristic odour of the seeds, an appreciable amount of a neutral oily substance,  $C_{18}H_{32}O_2$ , (b. p.  $214-215^{\circ}/18$  m.m., sp. gr. 0.9066 at  $16/16^{\circ}$ ,  $[\alpha]_D +42.4^{\circ}$ ), which is isomeric with chaulmoogric acid; this substance is being further investigated, as are also the seeds of *Gynocardia odorata* (R.Br.).

\*101. "The Constitution of Chaulmoogric Acid." Part I. By FREDERICK BELDING POWER and FRANK HOWORTH GORNALL.

With the object of ultimately determining the constitution of *chaulmoogric acid*,  $C_{18}H_{32}O_2$  (see preceding abstract), a number of its derivatives have been prepared and studied.

*Methyl chaulmoograte*,  $C_{17}H_{31}CO_2Me$  (m. p.  $22^{\circ}$ , b. p.  $227^{\circ}$  corr./20 m.m., sp. gr. 0.9119 at  $25/25^{\circ}$ ,  $[\alpha]_D +50^{\circ}$ , in chloroform), was prepared by the interaction of the acid, methyl alcohol, and hydrogen chloride. *Ethyl chaulmoograte*,  $C_{17}H_{31}CO_2Et$ , a colourless oil (b. p.  $230^{\circ}$  corr./20 m.m., sp. gr. 0.9079 at  $15/16^{\circ}$ ,  $[\alpha]_D +50^{\circ}$ ), was prepared in like manner. *Chaulmoogramide*,  $C_{17}H_{31}CO\cdot NH_2$  (m. p.  $106^{\circ}$ ,  $[\alpha]_D +57.3^{\circ}$  in chloroform), was obtained according to Aschan's method (Ber., 1898, xxxi., 2344). *Bromodihydrochaulmoogric acid*,  $C_{17}H_{33}BrCO_2H$  (m. p.  $36-38^{\circ}$ ; optically inactive), is formed when chaulmoogric acid is treated with hydrogen bromide in glacial acetic acid.

Ethyl chaulmoograte absorbs two atomic proportions of bromine in the cold, forming *ethyl dibromodihydrochaulmoograte*,  $C_{17}H_{31}Br_2CO_2Et$ , which is an oil.

When chaulmoogric acid is treated with sodium in boiling amyl alcohol, the ethylenic linking is not resolved, but there were obtained, after fractional distillation of the product, *chaulmoogryl alcohol*,  $C_{18}H_{33}OH$  (m. p.  $36^{\circ}$ ,  $[\alpha]_D +58.4^{\circ}$ ) and *chaulmoogryl chaulmoograte*,  $C_{17}H_{31}CO_2C_{18}H_{33}$  (m. p.  $42^{\circ}$ ), together with unchanged chaulmoogric acid.

The saturated acid, *dihydrochaulmoogric acid*,  $C_{17}H_{33}CO_2H$  (m. p.  $71-72^{\circ}$ , b. p.  $248^{\circ}/20$  m.m.; optically inactive), is formed, however, on reducing bromodihydrochaulmoogric acid with zinc dust and alcohol, or chaulmoogric acid with hydriodic acid and phosphorus. By the latter process, a hydrocarbon, *chaulmoogrene*,  $C_{18}H_{34}$  (b. p.  $193-194^{\circ}/20$  m.m.) is also formed. *Methyl dihydrochaulmoograte*,  $C_{17}H_{33}CO_2Me$  (m. p.  $26-27^{\circ}$ , b. p.  $222-223^{\circ}/20$  m.m.), was prepared from the corresponding acid.

Chaulmoogric acid is not attacked by fused caustic alkalis even at  $300^{\circ}$ .

When chaulmoogric acid was oxidised with cold permanganate (1 atom oxygen), *dihydroxydihydrochaulmoogric acid*,  $C_{17}H_{33}(OH)_2CO_2H$  (m. p.  $102^{\circ}$ ), was produced, but when the amount of permanganate was equivalent to 4-5 atomic proportions of oxygen, formic acid and two dibasic

acids were obtained, the latter having the formulae  $C_{15}H_{28}(CO_2H)_2$  and  $C_{15}H_{28}O(CO_2H)_2$  (m. p.  $128^{\circ}$ ). The *ethyl esters* of these acids were described.

The molecular magnetic rotation of ethyl chaulmoograte very closely approximates to the calculated value for an unsaturated ester of the formula  $C_{20}H_{36}O_2$ , having a closed ring and one ethylenic linking, the latter being contained in an allyl group. This conclusion, based on the magnetic rotation, is in harmony with the results obtained by the oxidation of the acid.

The further investigation of chaulmoogric acid is proceeding.

\*102. "Gynocardin, a New Cyanogenetic Glucoside." (Preliminary Note). By FREDERICK BELDING POWER and FRANK HOWORTH GORNALL.

In the course of an examination of the seeds of *Gynocardia odorata* (R.Br.), it was observed that when these were bruised and brought into water a strong odour of hydrogen cyanide is developed. This is due to the presence of a cyanogenetic glucoside, which the authors have isolated in a crystalline state, and designate *gynocardin*. It is very soluble in water, less freely in alcohol, and crystallises from these solvents in colourless needles which melt at  $161-162^{\circ}$  with slight decomposition, and have  $[\alpha]_D +19^{\circ} +37.1^{\circ}$ ; it gave on analysis the following percentages:—C=48.0; H=5.8; N=4.3. Its constitution is being determined.

\*103. "IsoNitrosocamphor." By MARTIN ONSLOW FORSTER.

An unstable *isonitrosocamphor* (m. p.  $114^{\circ}$ ), produced by the action of sodium and amyl nitrite on camphor, is converted into the yellow benzoyl derivative (m. p.  $105-106^{\circ}$ ) on treatment with benzoyl chloride in pyridine, whilst the colourless benzoyl derivative (m. p.  $136^{\circ}$ ) is produced in a similar manner from the stable *isonitrosocamphor* (m. p.  $152^{\circ}$ ).

Potassium ferrocyanide oxidises both forms to an unstable *peroxide*,  $C_{20}H_{28}O_4Na_2$ , which is insoluble in alkalis, and, the more fusible *isonitrosocamphor* being attacked first, the isomeride can be freed from it by using insufficient oxidising agent.

The *O-methyl ether* of *isonitrosocamphor*,  $C_{11}H_{17}O_2N$ , obtained by the action of methyl iodide in presence of silver oxide, crystallises in rectangular plates, melts at  $107^{\circ}$ , and has  $[\alpha]_D +197.7^{\circ}$ . Reduction converts it into amino-camphor, hydrolysis resolves it into  $\alpha$ -camphornitrilic acid, and hydroxylamine gives rise to the *oxime*,  $C_{11}H_{18}O_2N_2$ , which melts at  $188^{\circ}$  and has  $[\alpha]_D -95.3^{\circ}$ .

The *N-methyl ether* of *isonitrosocamphor*,  $C_{11}H_{17}O_2N$ , produced in association with the isomeride when methyl iodide acts on *isonitrosocamphor* dissolved in alcoholic potash, is a pale yellow oil which decomposes on distillation and has  $[\alpha]_D +279.8^{\circ}$ . Dilute acids and alkalis hydrolyse the substance to camphorquinone and  $\beta$ -methyl-hydroxylamine, reduction with zinc dust and acetic acid converting it into methylaminocamphor; hydroxylamine gives rise to *isonitrosocamphor* and the  $\beta$ -dioxime of camphorquinone, whilst phenylhydrazine transforms it into camphorquinonephenylhydrazine.

The decomposition of the colourless benzoyl derivative of *isonitrosocamphor* on exposure to light yields the anhydrides of benzoic and  $\alpha$ -camphornitrilic acids, the yellow isomeride giving rise to the same products. Determinations of molecular weight reveal no difference between the isomeric benzoyl derivatives.

The following derivatives of the camphorquinone dioximes have been examined:—The *acetyl*  $\alpha$ -dioxime (m. p.  $148-149^{\circ}$ ), the *dibenzoyl*  $\alpha$ -dioxime (m. p.  $153^{\circ}$ ), the *diacetyl*  $\beta$ -dioxime (m. p.  $119^{\circ}$ ), the *dibenzoyl*  $\beta$ -dioxime (m. p.  $189^{\circ}$ ), the *benzoyl*  $\gamma$ -dioxime (m. p.  $172^{\circ}$ ), and the *dibenzoyl*  $\gamma$ -dioxime (m. p.  $159^{\circ}$ ).

\*104. "The Basic Properties of Oxygen. Additive Compounds of the Halogen Hydrides and Organic Compounds, and the Higher Valencies of Oxygen, Asymmetric Oxy-

gen." By EBENEZER HENRY ARCHIBALD and DOUGLAS MCINTOSH.

An account was given of the preparation and properties of a number of compounds in which oxygen assumes a valency higher than 2.

The following compounds have been prepared and analysed:—

Acetone yields the hydriodide,  $(\text{CH}_3)_2\text{CO}\cdot\text{HI}$ ; the hydrobromide,  $(\text{CH}_3)_2\text{CO}\cdot\text{HBr}$ ; and the hydrochloride,  $(\text{CH}_3)_2\text{CO}\cdot\text{HCl}$ .

Ether gives rise to the compounds  $(\text{C}_2\text{H}_5)_2\text{O}\cdot\text{HI}$ ,  $(\text{C}_2\text{H}_5)_2\text{O}\cdot\text{HBr}$ , and  $(\text{C}_2\text{H}_5)_2\text{O}\cdot\text{HCl}$ .

Ethyl alcohol furnishes the following series:— $\text{C}_2\text{H}_5\cdot\text{OH}\cdot 2\text{HI}$ ,  $\text{C}_2\text{H}_5\cdot\text{OH}\cdot 2\text{HBr}$ , and  $\text{C}_2\text{H}_5\cdot\text{OH}\cdot 5\text{HCl}$ .

These products are white compounds, crystallising readily from their solutions in the liquefied halogen hydrides and melting at temperatures varying between  $-120^\circ$  and  $-9^\circ$ ; they are prepared by mixing together the organic compound previously cooled to  $-80^\circ$  with the appropriate halogen hydride in the liquid state, considerable quantities of heat being generated by the reaction.

A compound of anisole and hydrogen bromide has been prepared, which is probably phenylmethyloxonium bromide having the constitution  $\text{C}_6\text{H}_5\text{CH}_3\text{O}^+\text{Br}^-$  and therefore containing an asymmetric oxygen atom. This and similar compounds are at present under investigation.

105. "The Fermentation of the Indigo Plant." By CYRIL BERGTHEIL.

The fermentation of the indigo plant has been ascribed to a bacterium and also to enzymes, but has hitherto never been satisfactorily investigated. The author shows that, although there are many bacteria capable of producing the fermentation, it is in the main due to a specific enzyme occurring in the plant cells. A very active solution of this enzyme has been obtained, and the course of the fermentation produced by adding this to an extract of the indigo plant made in boiling water has been studied. It is found that the fermentation proceeds in a similar manner to that observed by Adrian Brown for invertase (*Trans.*, 1902, lxxxi., 273) and by Horace Brown and Glendinning for diastase (*Trans.*, 1902, lxxxi., 388), namely, that equal quantities of substance are transformed in equal intervals of time in the early stages, but not in the later ones. The point at which the course of the change can no longer be represented by a straight line is when 17 to 20 per cent of the total action has taken place. The curve representing the reaction ceases to be linear at about the same point when the time factor is kept constant, but the quantity of acting enzyme varied.

The optimum temperature for the action is almost exactly  $50^\circ$ , and the temperature at which the enzyme is destroyed is  $71^\circ$ . The rate of action is decreased by the presence of both acids and alkalis. Sodium acetate up to 1 per cent does not affect the rate of action. Various antiseptics all produced inhibition, formalin to the greatest, and boracic acid to the least extent. Emulsin can produce the indigo fermentation, but has a very weak action. It is doubtful whether the indigo enzyme can decompose amygdalin. Myrosin cannot ferment an extract of the indigo plant, and the indigo enzyme cannot ferment sinigrin. No evidence of the existence of an oxidase in the indigo plant was obtained.

106. "The Union of Hydrogen and Chlorine. Action of the Silent Discharge on Chlorine." By JOSEPH WILLIAM MELLOR.

By means of a special apparatus, chlorine can be subjected to any desired treatment before admixture with hydrogen without disturbing the equality of the proportions of hydrogen and chlorine in Bunsen and Roscoe's actinometer. The electrolytic gases are first sent through the liquid in the insolation vessel until a state of equilibrium is attained; the gases are then led into the insolation vessel without bubbling through this liquid at all.

If the chlorine gas is led through a glass "ozone apparatus" before admixture with hydrogen, the period of induction is curtailed, as indicated in the third column of the accompanying table. The numbers in the fourth column were obtained by the same apparatus, but the chlorine was exposed to the light of an acetylene flame before admixture with hydrogen. The numbers in the second column were obtained with chlorine not subjected to any previous treatment.

| Time, in minutes. | Ordinary chlorine. | Movement of index with—           |                                  |
|-------------------|--------------------|-----------------------------------|----------------------------------|
|                   |                    | 1. Chlorine and silent discharge. | 2. Chlorine and acetylene light. |
| 1                 | 0.1 c.m.           | 1.8 c.m.                          | 2.5 c.m.                         |
| 2                 | 0.1 "              | 4.4 "                             | 4.2 "                            |
| 3                 | 0.4 "              |                                   |                                  |
| 4                 | 1.5 "              |                                   |                                  |
| 5                 | 3.0 "              |                                   |                                  |
| 6                 | 4.0 "              |                                   |                                  |

(The asterisk denotes that the gases afterwards united at a constant rate).

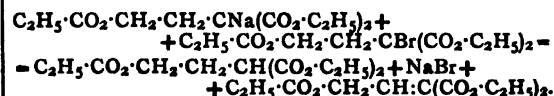
Moist chlorine is more chemically active towards hydrogen when subjected to—(1) A preliminary exposure to light; or to (2) a silent discharge of electricity.

Attempts are being made to isolate the active component.

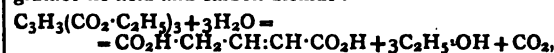
107. "Studies on Ethyl Carboxyglutarate. Part II. Action of Ethyl Bromocarboxyglutarate on Ethyl Sodiocarboxyglutarate. Formation of Ethyl Carboxyglutaconate." By OSWALD SILBERRAD and THOMAS HILL EASTERFIELD.

It was shown by the authors in the former part of the present work (*Proc.*, xx., 114) that ethyl sodiocarboxyglutarate is converted into haloid derivatives of ethyl carboxyglutarate by the action of halogens, no ethyl carboxyglutarate being formed. For this reason, the reaction between ethyl bromocarboxyglutarate and ethyl sodiocarboxyglutarate has been examined.

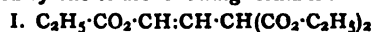
On bringing together the haloid and sodium derivatives of ethyl carboxyglutarate, they react and give rise to a mixture of the original ethyl carboxyglutarate and a new unsaturated ester,  $\text{C}_{12}\text{H}_{18}\text{O}_6$ . The reaction, which appears to be unlike anything hitherto observed, may be represented as follows:—



This unsaturated ester is thus to be regarded as ethyl  $\alpha$ -carboxy- $\Delta^{\beta}$ -glutaconate. On saponification, it yields a new carboxyglutaconic acid, which readily breaks up into glutaconic acid and carbon dioxide:—



from which it will be seen that its constitution must be represented by one of the following formulæ:—



or—



The latter of these formulæ must be the correct one, since the former represents ethyl isoconitate, from which the new isomeride differs in containing no active hydrogen and hence forming no sodium derivative.

108. "The Vapour Pressures of Liquid Mixtures of Restricted Mutual Solubility." (Preliminary Notice). By ARTHUR MARSHALL.

Although this subject has been somewhat fully treated from the theoretical side by Margules, Ostwald, Duhem, and others, yet with the exception of some somewhat rough measurements published by Konowaloff in 1881 and by Schrienemakers in 1890, no actual experiments have been made to test their conclusions. Experiments on mixture of water with ether, methyl acetate, and more especially

ethyl methyl ketone, have shown that in each case the curve for the total vapour pressure has much the same shape, but that this form is slightly different to those predicted by the foregoing authors.

When one of these comparatively volatile organic liquids is added gradually to water, the vapour pressure rises rapidly until the water becomes saturated. A second liquid phase then ensues and the curve becomes a horizontal straight line in accordance with the phase rule. When sufficient of the liquid has been added to dissolve the water completely, the curve again rises until it reaches a maximum, after which it falls again, until at 100 per cent it coincides with the vapour pressure of the pure substance. It is in this portion of the experimental curve that the variation from the predicted forms is noticeable.

In consequence of this peculiarity, the liquid mixture, unless containing a very large proportion of one or other of its constituents, will yield a distillate having a composition approximating closely to that which gives the maximum point. For example, with mixtures of methyl ethyl ketone and water, if the original liquid contains between 10 and 90 per cent of the ketone, the distillate will contain about 88.6 per cent of this compound.

109. "The Influence of Solvents on the Rotation of Optically Active Compounds. Part V. The Optical Activity of Certain Tartrates in Aqueous Solution." By THOMAS STEWART PATTERSON.

The rotations of sodium tartrate, potassium tartrate, potassium methyl tartrate, potassium ethyl tartrate, potassium *n*-propyl tartrate, methyl tartrate, ethyl tartrate, and *n*-propyl tartrate have been examined in aqueous solution at several concentrations and at temperatures between 10° and 100°.

In very dilute solution, sodium and potassium tartrates behave alike both as regards rotation value and influence of temperature-change, the rotation increasing as the temperature is raised.

It was shown that the rotations of the homogeneous compounds, could they exist in a superfused condition, would diminish on heating from 0° to 100°.

Of the three potassium alkyl tartrates, the *n*-propyl ester had the highest, and the methyl ester the least rotation, both in dilute solution and in the homogeneous state. Dilute solutions of these compounds all showed distinct temperatures of maximum rotation.

The rotations of the three alkyl tartrates were all raised by solution in water, and in dilute solution the rotations diminished rapidly on heating, this behaviour being just the opposite of that shown by the homogeneous esters.

The rotations in very dilute solution of the compounds examined were compared with each other and with that of the tartaryl ion.

The relationships existing among the experimental data were discussed with reference to the variations produced by solution and by change of temperature, and some suggestions, based on these observations, were advanced with respect to the temperatures at which the comparisons of rotation values may legitimately be instituted.

110. "The Nitration Products of the Isomeric Dichlorobenzenes." By PERCIVAL HARTLEY and JULIUS BEREND COHEN.

The progressive nitration of the dichlorotoluenes has been investigated by Cohen and Dakin (*Trans.*, 1902, lxxxi., 1344), and the results show that the two nitro-groups in the dinitro-derivatives of the six dichlorotoluenes follow strictly the meta-law of substitution.

Since the publication of the above, three papers describing the nitration products of *o*-, *m*-, and *p*-dichlorobenzenes have appeared (Morgan, *Trans.*, 1902, lxxxi., 1378; Blanksma and Terwogt, *Rec. Trav. Chim.*, 1902, xxi., 286; and Blanksma, *Ibid.*, 419).

From this it would appear that the principal products of nitration are an ortho-dinitro-compound in the case of *o*-dichlorobenzene, a meta-dinitro-compound in the case of *m*-dichlorobenzene, and a para-dinitro-compound in the

case of *p*-dichlorobenzene. The authors have repeated the work in order to ascertain quantitatively how far the divergence from the meta-law holds good.

They have been able to confirm the results of nitration of the *o*- and *m*-dichlorobenzenes, but not of the para-compound. The first gives with great difficulty an ortho-dinitro-compound, the second much more easily a meta-dinitro-compound, but *p*-dichlorobenzene gives about six times as much of the meta-dinitro-compound as of its paraisomeride. The only important exception to the meta-law is therefore afforded by *o*-dichlorobenzene.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 20, May 16, 1904.

Electrolysis of Calcium Chloride.—Henri Moissan.—In answer to M. Bullier's claim of priority of the electrolysis of calcium chloride, the author explains that his experiments differ from those of M. Bullier—(1) Because he has a liquid bath easily electrolysable; (2) because, even in the presence of carbon, more calcium than carbide is produced. In the author's first experiments he electrolyses a mixture of chloride and fluoride of calcium, and M. Bullier does not appear to have used such a bath at all.

The Density of Aqueous Saline Solutions considered as an Additive Property of the Ions. Existence of certain Hydrated Ions.—P. Vaillant.—Unsuitable for abstraction.

New Method of Exact Determination of the Molecular Weight of Permanent Gases. Atomic Weight of Hydrogen, Carbon, and Nitrogen.—Ph. A. Guye.—During numerical investigations on the fluid equation, the author establishes the fact that Van der Waals's equation leads to the relation  $V_m(1+a)(1-b)=R$ ; where  $V_m$  is the volume of a gram-molecule at 0° and 760 m.m.,  $a$  and  $b$  the two constants of the fluid equation, and  $R$  the constant of perfect gases (22.410.4). When applying this relation to the finding of the molecular weight of the so-called permanent gases (whose critical-point is below zero) the following modification is necessary:  $-V_m(1+a)(1-b)=R-mT_c$ ;  $m$  having the value 0.08473, the correction for  $mT_c$  always being very small. To determine the molecular weight deduced from gaseous densities the expression can be written  $M = \frac{L}{1000} \frac{R+mT_c}{(1+a)(1-b)}$ .

Preparation and Properties of Hypophosphorous Acid.—C. Marie.—The author describes a method of preparing hypophosphorous acid by a modification of Thomsen's method. When a large quantity is prepared, it is purified by fractional crystallisation, and on cold days the crystals can be filtered at the pump and left to dry for several days over phosphoric anhydride. Careful experiments show the fusion-point of this substance to be 26.5°. Hypophosphorous acid is decomposed by heat in the following manner:  $-2PO_2H_3 = PO_4H_3 + PH_3$ .

A Crystalline Chromous Tartrate.—G. Baugé.—The author finds that tartaric acid forms with chromium protoxide a crystalline anhydrous compound having a blue colour, and which is proved by analysis to have the formula  $C_4H_4CrO_6$ .

Triphenylmethane Colouring Matters.—Charles Lauth.—The author applies the reaction of replacing the hydrogen of the  $NH_2$  group in the aromatic amines by various substitutes to those leuco-bases of triphenylmethane which, after hydroxylation, sulphonation, and oxidation, give rise to very rich colouring matters

possessing the rare property of being unaltered by alkalis. Particularly, these substitutions were tried in leuco-derivatives which are obtained by the reduction of the condensation products of the essence of metanitrated bitter almonds on dimethyl- or diethylaniline. A series of substitution agents were made to act on these leuco-bases, and the products thus obtained examined from the point of view of the colouring matter which they form. The most interesting results are those given by the derivatives of acetyl chloride or acetic anhydride and the chloride of benzenesulphonic acid,  $C_6H_5SO_2Cl$ . Blue colouring matters are thus obtained analogous to patent blue, and possessing the same qualities of resistance towards alkalis, besides purity of shade. With the non-substituted amine derivative, greens are obtained.

**Preparation of  $\alpha\beta$ -Diketonic Ethers.**—L. Bouveault and A. Wahl.—The authors examine the constitution of certain phenylhydrazones and their relationship with the isomeric compounds resulting from the action of diazobenzene chloride on acetylacetic ether. They also propose to generalise this reaction, in order to arrive at the preparation of the  $\alpha\beta$ -diketonic ethers,  $R-CO-CO-COOC_2H_5$ .

**Action of  $PCl_3$  on certain Cyclic Primary Amines at Boiling-point.** Reduction of  $PCl_3$  with Formation of Phosphorus.—P. Lemoult.—The author finds that when  $PCl_3$  reacts on the cyclic amines,  $RNH_2$ , at their boiling-point, there are formed, besides the orange compound described in a former paper, the same products which  $PCl_3$  gives without a gaseous evolution, but with production of white phosphorus.

**New Polymers of Formaldehyde.**—A. Seyewetz and M. Gibello.—Up to the present time four polymers of formaldehyde have been described. The author prepares other polymers having absolutely different properties, of which a list is given.

**Action of Paraformaldehyde on the Sesquiterpenes.** P. Genvresse.—MM. Ladenburg and Kriewitz examined the action of paraformaldehyde on certain terpenes in sealed tubes, and they obtained addition products with alcoholic function. The author finds that the sesquiterpenes behave in the same manner with trioxymethylene, but the action is not quite so easy and the yields smaller. His experiments were chiefly with a view to obtaining perfumes, and he shows that caryophyllene, clovene, and cadinene will combine molecule for molecule with formic aldehyde.

*Bulletin de la Société Chimique de Paris.*

Series 3, Vol. xxix., Nos. 18—19.

**Normal Existence of Arsenic in all the Organs of the Animal Economy.**—Armand Gautier.—The author's experiments show that arsenic exists normally in certain tissues, animal and vegetable, and that it is absent, or in unappreciable quantities, in many others. He is not yet satisfied—as some writers are—that this metalloid is present in all living cells.

**Electrolytic Separations.** 1. Manganese and Iron. 2. Aluminium and Iron, or Nickel; Zinc and Iron.—MM. Hollard and Bertiaux.—Already noticed.

**Estimation of the Persulphates.**—C. Marie and L. J. Brunel.—Already inserted in full.

**Ratio between the Solubility of Lime in the presence of Alkalis, and the Caustification of the Alkaline Carbonates.**—Alex. d'Anselme.—The author finds that the solubility of lime in water diminishes sensibly when the temperature is increased; it also diminishes when the temperature is constant if the proportion of caustic soda is augmented. With a constant quantity of caustic soda, the solubility diminishes when the temperature rises.

**Examples of the Complexity of the Catalytic Action of Metals: Vivifying and Paralysing Influences.**—M. Trillat.—Already noticed.

**New Method for the Attack of Galenas and Chalcopyrites.**—C. Boucher.—Already inserted in full.

**Manner of Division of Mixed Organo-magnesian Compounds.** Action of Oxide of Ethylene.—V. Grignard.—Already noticed.

**Action of Chloride of Ethyloxalyl on the Organo-magnesian Compounds.**—V. Grignard.—Already noticed.

**Primary Phenylethyl Alcohol.**—V. Grignard.—This alcohol was obtained by the action of commercial polyoxymethylene on benzylchloride of magnesium, according to the method already discovered by the author in collaboration with M. Tissier. The primary phenylethyl alcohol thus obtained has a fairly agreeable aromatic odour; it crystallises, by cooling under a current of water, in long needles fusible at  $33^\circ$ ; its density taken at surfusion at  $15.2^\circ = 1.0389$ .

**Method for the Preparation of Diethylacetacetate of Methyl.**—V. Grignard.—Dissolve 11.5 grms. of sodium in 70 grms. of methanol, add half a molecule of acetacetic ether, half a molecule of iodide of ethyl, and heat (with a reflux condenser) for two hours. When the alkalinity has almost completely disappeared, distil a portion of the methyl alcohol on the water-bath, treat the residue with water, and then in the usual manner. The product obtained separates into two portions by fractional distillation. The most important boils at  $187-190^\circ$  under 750 m.m.; this is the ethylacetacetate of methyl. The other is the corresponding ethylic ether. These were treated separately with iodide of ethyl and methylate of sodium in methyl solution. In both cases nothing but diethylacetacetate of methyl was obtained.

**Preparation of Nitric Ethers.**—L. Bouveault and A. Wahl.—Already noticed.

**Preparation of the Nitrous Ethers of Alcohols.**—L. Bouveault and A. Wahl.—Already noticed.

**Isonitrosomalonic Ethers.**—L. Bouveault and A. Wahl.—Already noticed.

**Action of Chloracetimide on some Aromatic Amines.**—A. L. Lumière and F. Perrin.—The authors have obtained a series of nine new compounds by reacting with chloracetimide on an amide of the type  $R-NH_2$  or  $R'>NH$ , a molecule of hydrochloric acid is eliminated, and the radical  $-CH_2-CO-NH_2$  becomes joined to the group  $NH$  of the amine.

**Action of Phenylhydrazine on the Alcoholic Bromides and Iodides.**—J. Allain-le Canu.—Already noticed.

**Cyclohexane and its Chlorised Derivatives.**—P. Sabatier and A. Mailhe.—The physical properties of the cyclohexane  $C_6H_{12}$  are identical with those of the carbide prepared synthetically from pimelic acid by Zéliniski. The existence of the aromatic nucleus in this carbide was proved by the action of bromine, which changes it into tetrabromobenzene. It is also shown by the reactions of the chlorised derivatives, which are fully described in this paper.

**Separation of the Principles Decomposing Peroxide of Hydrogen contained in the Hæmatin.**—J. Ville and J. Moitessier.—The authors have obtained a ferment from blood, and show that it is the fixation of this ferment on the fibrin that is the essential, if not the exclusive, cause of the decomposing action of this proteic principle on peroxide of hydrogen.

**Influence of the Nature of the Surroundings on the Formation and Evolution of the Odorous Compounds in Plants.**—E. Charabot and A. Hebert.—A long paper, not suitable for abstraction.

**A New Gas Burner.**—L. Quennessen.—Already noticed.

## MISCELLANEOUS.

The Absorption Spectra of Solutions of Salts of Didymium containing Phosphoric Acid, and on Orthophosphate of Didymium.—A. Waegner.—The addition of  $\text{PO}_4\text{H}_3$  in excess to a solution of chloride of didymium gives a fairly stable limpid solution; its absorption spectrum is not the same as that of the primitive salt. The bands are decidedly displaced, and especially those near the D line, which are replaced by a large number of narrower bands (see plate in the original). On the addition of water, or by increasing the temperature, the solution gives a precipitate of phosphate of didymium; the best way is to pass a strong current of steam through the solution. A pinkish-white amorphous precipitate is formed, becoming quite white on calcination, attackable only by concentrated acids after calcination. This salt is  $\text{PO}_4\text{Di}_2\text{H}_2\text{O}$  (at  $100^\circ$ ). It gives a very characteristic emission spectrum. The anhydrous  $\text{PO}_4\text{Di}$ , treated with concentrated  $\text{SO}_4\text{H}_2$ , becomes heated, and is partially dissolved; if we evaporate strongly to drive off the excess of  $\text{SO}_4\text{H}_2$ , there remains a residue of sulphate and metaphosphate of didymium.—*Berichte*, vol. xxxvi., p. 3055.

Examination of the Methods for the Estimation of Antimony.—L. A. Youtz.—The boiling-points of the chlorides of antimony, arsenic, and tin are very different ( $230^\circ$ ,  $134^\circ$ , and  $114^\circ$ ). On the other hand, they volatilise from their hydrochloric acid solutions at totally different temperatures. Tin (as  $\text{SnCl}_4$ ) keeps nearest to its boiling-point,  $114^\circ$ , but antimony and arsenic commence to volatilise respectively at  $108^\circ$  and  $75-80^\circ$ . Whether the antimony is in solution in the antimonious or the antimonim form, its volatility is the same. As the chloride,  $\text{SbCl}_3$ , prepared from Cl and Sb, is much more volatile than the trichloride, we are inclined to admit that the latter is hydrolysed in solution in  $\text{HCl} + \text{SbO}_4\text{H}_3$ . It should be the same in solutions in which the antimony has been dissolved by a mixture of  $\text{NO}_3\text{H}$  and  $\text{ClO}_3\text{K}$ . Further, we cannot attribute this slight volatility to the formation of double salts, for the same results are observed by oxidising with a mixture of  $\text{NO}_3\text{H} + \text{HCl}$ . This facility for hydrolysing is also observed with all the elements of the group, and becomes fainter from P to Bi.—*Zeit. Anorg. Chem.*, vol. xxxv., p. 55.

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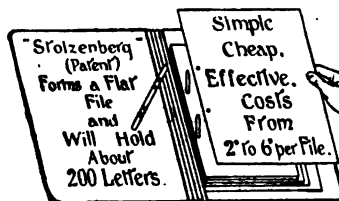
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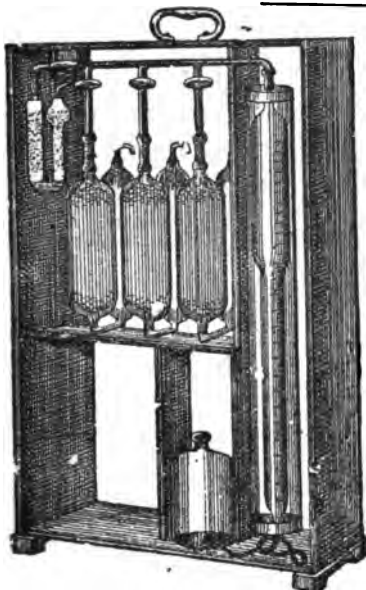
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### CONTENTS.

| ARTICLES:—                                                                                                      | PAGE |
|-----------------------------------------------------------------------------------------------------------------|------|
| The Spectrum of the Radium Emanation, by Sir William Ramsay, K.C.B., F.R.S., and Prof. J. Norman Collie, F.R.S. | 301  |
| A Volumetric Method for the Estimation of Mercury Fulminate, by Henry W. Brownson, B.Sc., Ph.D.                 | 303  |
| A New Magnesia Cupel                                                                                            | 304  |
| London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.                                    | 304  |
| International Atomic Weights, by Joji Sakurai and K. Ikeda                                                      | 305  |
| PROCEEDINGS OF SOCIETIES—                                                                                       |      |
| PHYSICAL SOCIETY                                                                                                | 306  |
| THE FARADAY SOCIETY                                                                                             | 307  |
| ROYAL SOCIETY.—Conversations at Burlington House                                                                | 308  |
| NOTICES OF BOOKS                                                                                                | 309  |
| OBITUARY.—Thomas Bayley                                                                                         | 310  |
| CHEMICAL NOTICES FROM FOREIGN SOURCES                                                                           | 310  |
| MISCELLANEOUS                                                                                                   | 310  |

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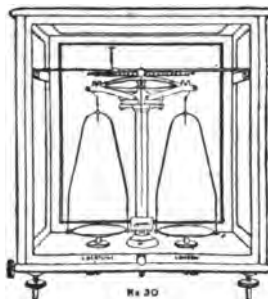
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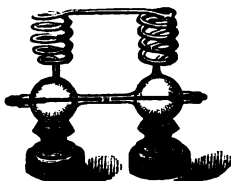
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# THE CHEMICAL NEWS.

Vol. LXXXIX., No. 2326.

## THE SPECTRUM OF THE RADIIUM EMANATION.\*

By Sir WILLIAM RAMSAY, K.C.B., F.R.S.,  
and  
Prof. J. NORMAN COLLIE, F.R.S.

ATTEMPTS have been made since July, 1903, to see and map the spectrum of the emanation from radium, for at that date the conversion of the emanation into helium was observed by Ramsay and Soddy; and during the first discharge of the induction current through the emanation, it was believed that a peculiar spectrum was noticed; indeed, three lines were persistent, and were mentioned in the communication on the subject in the *Proceedings*.

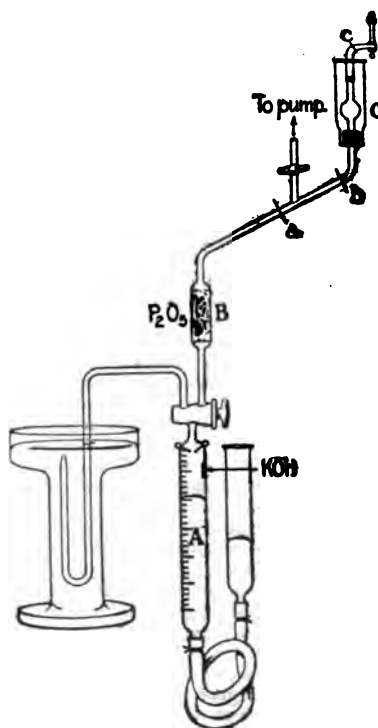
But such attempts have uniformly failed; at the first moment of the discharge, indeed, a brilliant spectrum has twice been observed, which soon became confused and indistinct. It faded before it was possible to map it, and owing to the presence of impurities, generally carbon monoxide, nitrogen, or hydrogen, the special spectrum was obscured. All that could be said was that it appeared to present some brilliantly green lines.

These experiments, however, have not been fruitless; they have led to better knowledge of the precautions which it is necessary to take to eliminate impurities. The arrangement of the apparatus, too, has been simplified, and the manipulation made easier. As it is possible that others may wish to repeat the experiments, and may perhaps have even better success in mapping the spectrum, we think it well to enter into the details of the manipulation somewhat minutely, and to give a woodcut of the apparatus employed.

The stock of radium bromide (about 109 m.grms.) dissolved in about 10 c.c. of water in two small bulbs was attached by sealing to a small Töpler's pump. Between the pump and the bulb there was a stop-cock, greased of course, to ensure freedom from leakage, but in order to prevent the long contact of the emanation with the stop-cock, and its possible contamination with carbon dioxide, the mercury from the pump was caused to flow past the stop-cock by raising the reservoir of the pump and closing the exit tube at its lower end; the mercury slowly leaked past the valve of the pump, passed the tap (which was then shut); and so confined the space above the radium bromide by means of mercury. As radium bromide yields electrolytic gas, containing an excess of hydrogen, the pressure gradually rose; the mercury in contact with this gas remained perfectly bright, and showed no tendency to adhere to the glass; the presence of ozone thus appears to be excluded, but this excess of hydrogen will form the subject of a future communication.

The emanation was allowed to accumulate for fourteen days. The pump was exhausted until no trace of a bubble passed down the capillary exit tube. But as even then a trace of air must have remained in the barrel, the tap leading to the bulbs containing the radium bromide was turned rapidly, so as to admit a trace of the electrolytic gas into the pump and "wash it out." This gas was rejected. The remaining electrolytic gas with the emanation was collected in a tube which had previously been heated to redness, and then twice washed out with pure oxygen. The mercury in the collecting tube was then boiled, and the bubble of gas removed. It was hoped thereby to have eliminated every trace of nitrogen. The gas was then introduced into the gas-burette, shown in the figure, through

the inverted syphon. All the mercury was freshly filtered and pure. The apparatus, too, was freshly constructed and heated to redness to burn out traces of dust. The gas-burette had been washed out with alkali and with nitric acid, and then with a stream of distilled water; it was dried by drawing through it a stream of dust-free air. Some slightly moist caustic potash was melted on to the glass, near the sparking wires; this was intended to absorb any trace of carbon dioxide which might have chanced to be formed during the explosion of the electrolytic gas by the burning of dust. The rubber tube was cemented on to the burette; the burette was washed out twice with oxygen, and by lowering the reservoir several times the upper end was made a torricellian vacuum; it was left thus for some time, so as to ensure the removal of adhering nitrogen from the walls of the tube.



The electrolytic gas was then introduced, and exploded. As the explosion-burette was graduated, the total volume of the gas, as well as that of the residual hydrogen, was read. There were 16.43 c.c. of gas; the residual hydrogen measured 1.01 c.c. at normal temperature and pressure, and thus amounted to 6.18 per cent of the total. The volume of this gas was increased by lowering the pressure so that it was in contact with the fused potash. It was left for more than an hour; the potash, of course, was wet with the water formed by the explosion.

The capillary tubes above the stop-cock of the gas-burette, which had been twice washed out with oxygen, were pumped as empty as possible, until the vacuum-tube showed only the yellow and green lines of the mercury spectrum and the faintest trace of a hydrogen spectrum. A strong current was passed between the electrodes so as to heat them and expel occluded oxygen. After this process had been repeated as long as was thought safe, until, as remarked, the hydrogen spectrum was extremely faint, the tap to the pump was closed. The hydrogen containing the emanation was then admitted from the explosion-burette; it was dried by passage through the narrow tube *a* filled with phosphoric anhydride, and it entered the bulb *c*,

\* A Paper read before the Royal Society, May 19, 1904.

and the vacuum-tube D. This vacuum-tube was made of lead-glass, with electrodes of aluminium. It was 2.5 c.m. long, with a capillary of about 1 c.m. in length. The aluminium electrodes were closely surrounded with glass, fused round them, so as to limit the capacity of the tube as much as possible; it was probably under one-twentieth of a c.c.

Liquid air was next poured into the jacket surrounding the bulb C, and the reservoir was raised and lowered half a dozen times, so as to convey all the gas into contact with the cooled bulb. The mercury was then raised to the level *a*, and the tap to the pump opened; and while the jacket was kept replenished with liquid air the hydrogen was pumped off until its spectrum had almost entirely disappeared, the red line being hardly visible. The tap to the pump was then closed, the level of the mercury was raised to *b*, and the liquid air allowed to evaporate. The bulb was so bright that it was easy to read the time on a watch. The mercury was then raised to the level *c*, and the current passed. The spectrum was very brilliant, consisting of very bright lines, the spaces between them being perfectly dark; it had a striking resemblance in general character to the spectra of the gases of the argon group.

A direct-vision spectroscopic, made to special design by Heele, with an illuminated scale for reading, had immediately before been standardised by noting the position of the leading lines of helium and hydrogen. They were found to lie exactly on a scale which had previously been constructed. The new lines were read as rapidly as possible, an operation which required about half a minute. During a second reading many of the lines had faded, and the secondary spectrum of hydrogen began to appear, and rapidly grew stronger. It was identified by throwing into a field a hydrogen spectrum through the small prism; and it soon became so powerful as to mask the spectrum of the emanation completely. In order to attempt to recover it, the mercury was again drawn down to *a*, and liquid air again poured into the jacket; the emanation again condensed, and the tap to the pump was opened, and the hydrogen removed by the pump until its spectrum was again hardly visible. On repeating the series of operations already described, the spectrum of the emanation was seen a second time, but it was so transient that only the position of some of the lines could be confirmed.

Next day, only the spectrum of hydrogen was visible; its secondary spectrum was strong. The day after, the same was the case; but interposing a jar and spark-gap brought out two lines which had previously been mapped; they were very feeble.

In the table which follows, all the strong lines which were read are given; the degree of coincidence of those which are of known wave-length shows the approach to accuracy obtained; the error is probably less than five Angström atoms.

| Wave-length. | Remarks.                                                                                                      |
|--------------|---------------------------------------------------------------------------------------------------------------|
| 6567         | Hydrogen C; true wave length, 6563; observed each time.                                                       |
| 6307         | Observed only at first; evanescent.                                                                           |
| 5975         | " " "                                                                                                         |
| 5955         | " " "                                                                                                         |
| 5805         | Observed each time; persistent.                                                                               |
| 5790         | Mercury; true wave-length, 5790                                                                               |
| 5768         | " " 5769                                                                                                      |
| 5725         | Observed only at first; evanescent.                                                                           |
| 5595         | Observed each time; persistent and strong.                                                                    |
| 5465         | Mercury; true wave-length, 5461.                                                                              |
| 5105         | Not observed at first; appeared after some seconds; persisted, and was visible during the second examination. |
| 4985         | Observed each time; persistent and strong.                                                                    |
| 4865         | Hydrogen F; true wave-length, 4861.                                                                           |
| 4690         | Observed only at first.                                                                                       |
| 4650         | Not observed when the emanation was examined again.                                                           |
| 4630         | Ditto.                                                                                                        |
| 4360         | Mercury; true wave-length 4359.                                                                               |

When the spectrum was examined two days later, besides the hydrogen and mercury lines, there were seen:—5595, feeble; 5105, feeble; 4985, very feeble; this was with a jar and spark-gap interposed; the ordinary discharge showed only the primary and secondary spectra of hydrogen, and that of mercury.

Eleven days later, the emanation from the same stock of radium bromide was collected, and treated in exactly the same manner. This time, however, an excess of conscientiousness made us continue to extract gas with the pump from the bulb C containing the frozen emanation, surrounded by liquid air, for too long a time. Every two or three strokes of the pump collected a minute bubble, occupying about the tenth of a millimetre in length of the very narrow fall-tube of the pump, which was really a fine-bore capillary. The yield of gas appeared to be continuous; and when these bubbles were examined in the dark they were brilliantly luminous. This gas was really the emanation, which possesses a feeble vapour pressure even at the temperature of liquid air. Needless to say, on attempting to examine the spectrum, little was seen, for the pressure of gas in the vacuum-tube was too low.

The tube was therefore washed out with the gas which had been pumped off, and the process was repeated. The minute bubbles which passed down the capillary fall-tube of the pump were examined, and pumping was stopped when they showed a very faint luminosity in the dark. On compressing the emanation into the spectrum-tube, the spectrum was again brilliant, and measurements were made. It was found possible to read the lines several times, for although the spectrum faded in less than a minute, it appeared to recover on ceasing to pass the current. But this recovery soon failed, and, as before, nothing could be detected after five minutes but the primary and secondary spectra of hydrogen. Now the tube was practically vacuum before warming the bulb containing the emanation; no current would pass; but it is, of course, possible that the gas carrying the emanation had not been perfectly dried in passing through the tube B, containing phosphoric anhydride; any water-vapour would have condensed in the cooled bulb C, and would only slowly have vaporised into the vacuum-tube. On arriving there, it would give the hydrogen spectrum. Another possibility is that it may have come out of the electrodes; for it has been frequently noticed in glowing out a vacuum-tube with aluminium electrodes, that even after all trace of hydrogen has been removed by passing the discharge so as to heat the electrodes, and by pumping, the hydrogen spectrum has re-appeared on admitting a trace of one of the gases of the argon group, and passing the discharge for a longer time; but the intensity of the spectrum which replaced that of the emanation may perhaps warrant the supposition that hydrogen as well as helium is one of the products of the disintegration of the emanation. This, however, is very doubtful, and judgment may be suspended until more satisfactory evidence is forthcoming.

The lines read were:—

| Wave-length. | Remarks.                                                                |
|--------------|-------------------------------------------------------------------------|
| 6359         | Not observed before; faint.                                             |
| 5975         | Observed before; faint.                                                 |
| 5955         | " " "                                                                   |
| 5890         | Not observed before; faint.                                             |
| 5854         | " " "                                                                   |
| 5725         | Observed before; fairly strong.                                         |
| 5686         | Not observed before; faint                                              |
| 5595         | Observed before; strong and persistent.                                 |
| 5580         | Not observed before; faint.                                             |
| 5430         | " " "                                                                   |
| 5393         | " " "                                                                   |
| 5105         | Bright; persistent; observed before.                                    |
| 4985         | " " "                                                                   |
| 4966         | Not observed before; "bright," but transitory.                          |
| 4640         | Transitory; possibly 4650 and 4630, were seen before as distinct lines. |

The line 4966 was particularly brilliant at first; but it soon assumed secondary importance. Some lines which had previously been observed were not seen; they are 6307, 5805, 5137, and 4690. An attempt was made to obtain the spectrum with a jar and spark-gap; but only hydrogen and mercury were to be seen. The resistance soon became very high, and there was danger of piercing the vacuum-tube.

Previous attempts in conjunction with Mr. Soddy gave lines with wave-length 5725 (jar), 5595 (no jar), 5105 (no jar), 4985 (no jar); the line 5585 was observed three times, and 5105 twice previously. The lines 6145 and 5675 mentioned in our last paper (April, 1904) were not seen unless the latter is identical with 5580. It may perhaps be mentioned that the line 5595 was seen by Pickering in the spectrum of lightning, and was not identified with a line in the spectrum of any known gas; it is said to have been a very strong line, of intensity 30 (*Astrophysical Journal*, 1901, vol. xiv., p. 368).

There can be no doubt that the lines given are the chief lines in the visible spectrum of the emanation; as for the pressure, the volume of emanation was about 1/30,000th of a c.c., and the capacity of the vacuum-tube, say, 1/20th; this would make the pressure about 1/10th of a millimetre. It may have been twice as much, for the numbers given are merely estimates.

It may be remembered that, at the Chemical Congress held in Paris in 1900, it was suggested that no element should receive a name until its spectrum had been mapped. Of course, the converse does not follow that, after the spectrum of an element has been mapped, it should receive a name. The "emanation from radium," however, is a cumbersome expression, and sufficient evidence has now been accumulated that it is an element, accepting that word in the usual sense. It is true that it is only a transient element, and ought in justice to be called a compound; but of what? It stands on a wholly different plane to any known compound in the amount of heat with which it parts during its spontaneous change, and in the peculiar electrical phenomena which accompany its transformation. It is a gas; it follows Boyle's law; as Rutherford and Soddy have shown, it resembles the gases of the argon series in its indifference to chemical reagents, for it not merely withstands the prolonged action of magnesium-lime, at a red heat, but also, as Ramsay and Soddy have proved, prolonged sparking with oxygen in presence of caustic potash. Its molecular weight has been found to be nearly 200, and, if it is monatomic, that number would also express its approximate atomic weight. Now, it appears advisable to devise a name which should recall its source, and, at the same time, by its termination, express the radical difference which undoubtedly exists between it and other elements. As it is derived from radium, why not name it simply "extradio"? Should it be found that the emanation, which is supposed to be evolved from thorium, is really due to that element, and not to some other element mixed with thorium in exceedingly small amount, a similar name could be given, namely, "exthorio." If the existence of actinium as a definite element is established, its emanation would appropriately be named "exactinio." It is unlikely that others will be discovered, but, if they are, the same principle of nomenclature might be applied.

It should be stated, in conclusion, that Mr. Soddy collaborated in the experiments preliminary to this successful mapping of the spectrum; had he not been obliged to leave England, he would, no doubt, have shared whatever credit may attach to this work.

**Technological and Scientific Dictionary.**—Part III. of this Dictionary has just been issued by the publishers, George Newnes, Ltd. We have previously referred to the merits of this work, of which the present number comprises those subjects coming between C O P (Copper losses, *Elect. Eng.*), and E L E (Electric lighting), the pages running from p. 129 to p. 192.

## A VOLUMETRIC METHOD FOR THE ESTIMATION OF MERCURY FULMINATE.

By HENRY W. BROWNSDON, B.Sc., Ph.D.

THE use of sodium thiosulphate solution as an agent for decomposing fulminate of mercury was first brought to my notice by Mr. George Smith, manager of Nobel's Fulminate Factory at Polmont, and with a view to its adoption in this laboratory I have had the opportunity of making an inquiry into the nature of the reaction. My work in this direction is, however, still incomplete, and I now only wish to mention an analytical application which has resulted as a side issue.

When fulminate of mercury is decomposed by excess of sodium thiosulphate solution, the resulting solution reacts alkaline, and the determination of the amount of alkali thus formed affords an easy and quick volumetric method for the estimation of mercury fulminate.

**Method.**—The only solution required is one of sulphuric acid approximately N/10. This is standardised as follows:

0.04 to 0.05 grm. of pure mercury fulminate—prepared by dissolving the ordinary commercial product (1 mol.) in pure potassium cyanide (1 mol.) solution, precipitating the mercury fulminate from the solution of the double salt by means of dilute nitric acid, washing several times by decantation, and finally on the filter-paper till quite free from acid, and then drying at 80 to 90°—is weighed out into a 100 c.c. flask containing about 50 c.c. water; 1 grm. of pure sodium thiosulphate is then added, the flask well shaken till all the fulminate has dissolved, and then made up to the 100 c.c. mark. Portions of 25 c.c. are withdrawn by means of a 25 c.c. pipette, and titrated with acid after addition of one drop methyl orange. It is well to allow the acid to drop from the burette drop by drop, shaking the liquid to be titrated all the time, so that no portion of the liquid becomes locally acid.

The above is repeated on three separate weighings, and the mean taken. The value of 1 c.c.  $H_2SO_4$  in grms. of fulminate is thus obtained.

As applied to the determination of mercury fulminate in cap or detonator compositions, one precaution is necessary, viz., that the amount of fulminate in the weighed out portion of composition should not exceed 0.05 grm., otherwise the results will tend to be low on account of the alkali set free reacting with the finely divided antimony sulphide. For a cap composition containing 15 to 20 per cent fulminate, a weighing of 0.25 grm. is suitable, whilst for a detonator composition containing 35 to 40 per cent fulminate a weighing of 0.12 grm. is sufficient.

The above amount of either cap or detonator composition is weighed out into a 100 c.c. flask containing about 50 c.c. water, 1 grm. of pure sodium thiosulphate added, and the contents well agitated till one is sure all the fulminate has been decomposed (one and a-half to two minutes). The flask is then filled up to the 100 c.c. mark, the contents mixed, and filtered through a dry folded filter-paper into a dry flask. The first runnings are returned through the filter-paper till the filtrate is clear. Portions of 25 c.c. are then titrated as above. To ensure accuracy, a rough preliminary experiment is made, and then two subsequent determinations on two separate weighings, taking the mean of the two latter for the result.

The solutions must not be allowed to stand, but must be titrated as soon as possible after the addition of the thiosulphate, since after a time secondary reactions set in which neutralise some of the alkali giving low results.

Following are a few typical results obtained by applying this method to compositions containing a known weight of fulminate.

By careful working it is possible to estimate the weight of fulminate to within  $\pm 0.0001$  grm., an error which is less than 0.1 per cent when calculated out for the percentage of fulminate in the composition. I am continuing this work, and hope shortly to give a fuller account of the reaction

| Weight of fulminate taken. | Weight of $\text{Sb}_2\text{S}_3$ . | Weight of $\text{KClO}_3$ . | $\text{H}_2\text{SO}_4$ (1 c.c. = 0.00832 grm. fulminate). |                   | Weight of fulminate found. |
|----------------------------|-------------------------------------|-----------------------------|------------------------------------------------------------|-------------------|----------------------------|
|                            |                                     |                             | First titration.                                           | Second titration. |                            |
| Grm.                       | Grm.                                | Grm.                        | C.c.                                                       | C.c.              | Grm.                       |
| 0.0328                     | 0.05                                | 0.05                        | 1.00                                                       | 0.98              | 0.0329                     |
| 0.0483                     | 0.05                                | 0.05                        | 1.45                                                       | 1.45              | 0.0482                     |
| 0.0462                     | 0.05                                | 0.05                        | 1.40                                                       | 1.38              | 0.0462                     |
| 0.0425                     | 0.10                                | 0.10                        | 1.25                                                       | 1.30              | 0.0424                     |
| 0.0458                     | 0.10                                | 0.10                        | 1.35                                                       | 1.40              | 0.0457                     |

along with its bearing on the composition of the fulminates, a point on which the decomposition by thiosulphate and by sodium sulphide seems to throw much light.

I have much pleasure in acknowledging here the kindness of the directors of the Kings Norton Metal Company, Limited, who have placed facilities at my disposal for carrying out this work.

### A NEW MAGNESIA CUPEL.

THOSE of our readers who employ magnesia in preference to the bone-ash cupels which have for so long held the field, will be interested in an ingenious improvement which has recently been made in the Morganite cupels introduced by the Morgan Crucible Company, of Battersea.

Although the numerous advantages possessed by magnesia cupels as compared with those of bone-ash are now generally recognised, the slightly slower absorption of the litharge by the former tends to permit a portion of the litharge to soak down through the bottom before it can diffuse into the body of the cupel. Hence, before the cupels are fully saturated, a portion of the litharge is lost, and the muffle may become corroded by the litharge. The former is usually of little consequence, and the latter may be obviated by the practice which many assayers adopt, of keeping a layer of bone-ash, magnesia, or other absorbent in the muffle, but both are practically abolished by use of the new cupel.

The improvement, as shown in the accompanying sketch, consists merely in making the cupel with a cup-shaped depression in the bottom; in fact, in making it double ended. The litharge soaks through exactly as in



ordinary cupels, but as the middle portion is not in contact with the muffle floor, it has time to diffuse into the body of the cupel before it accumulates sufficiently to actually escape at the bottom.

This simple device has been found to answer all expectations. It permits of the cupellation of a larger quantity of lead per cupel without loss of litharge, and it is stated by the inventors that cupellation progresses more rapidly in these than in the old form of cupel.

As we presume that these cupels will not cost more than the old form, it may be worth while for our readers to try them and ascertain how far their experience bears out the statements of the inventors as to the results which they have obtained in practice.

### LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MAY 31ST, 1904.

By SIR WILLIAM CROOKES, F.R.S.,  
and  
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, ESQ., M.Inst.C.E.,  
*Water Examiner, Metropolitan Water Act, 1871.*

London, June 8th, 1904.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 204 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from May 1st to May 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 204 samples examined by us during the month, all were clear, bright, and well filtered.

There has been a large excess of rain during the month of May. The actual rainfall measured at Oxford during the month was 3.20 inches, and the average fall is 1.75 inches; thus we have an excess of 1.45 inches, which, if added to the previous excess of 0.98 inch, makes a total of 2.43 inches, or 27.9 per cent above the average for thirty-five years.

Our bacteriological examinations of 370 samples taken during the month have given the results recorded in the following table. Besides these samples we have examined 364 others from special wells, standpipes, &c., making 734 samples in all:—

|                                                                                                           | Microbes per c.c. |
|-----------------------------------------------------------------------------------------------------------|-------------------|
| New River, unfiltered (mean of 25 samples) ..                                                             | 116               |
| New River, filtered (mean of 74 samples) ..                                                               | 4                 |
| Thames, unfiltered (mean of 25 samples) ..                                                                | 4120              |
| Thames-derived water from the clear-water wells of eight Thames-derived supplies (mean of 196 samples) .. | 15                |
| Ditto ditto .. .. . highest                                                                               | 181               |
| Ditto ditto .. .. . lowest                                                                                | 9                 |
| River Lea, unfiltered (mean of 25 samples) ..                                                             | 225               |
| River Lea, from the East London Water Company's clear-water wells (mean of 25 samples) ..                 | 19                |

Of the 295 daily samples taken from the general filter wells of the Metropolitan Water Companies, twenty-two samples, or 7.4 per cent, were sterile. Five samples, or 1.6 per cent, contained more than 100 microbes, and of these, two samples contained more than 150 microbes per c.c. The five excess samples contained an average of 143 microbes per c.c. In April five excess samples contained an average of 133 microbes per c.c.

From the above figures it will be seen that the high condition of purity observed during April has been well maintained during the month of May, in spite of the great increase of rainfall.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.  
JAMES DEWAR.



INTERNATIONAL ATOMIC WEIGHTS.\*

By JOJI SAKURAI and K. IKEDA.

Science College, Imperial University,  
Tokyo, Japan, May 19, 1904.

To the Editor of the *Chemical News*.

DEAR SIR,—I am sending to you by this mail a copy of the reprints of a letter addressed by my colleague Prof. Ikeda and myself to Professor F. W. Clarke on the subject of International Atomic Weights. Although the letter, which forms a part of our report to the Tokyo Chemical Society on International Atomic Weights, has been printed in the *Journal* of that Society, I shall be obliged if you can find space for its publication in your valuable journal, knowing that hundreds of the readers of the *CHEMICAL NEWS* take interest in the subject, and that the *Journal of the Tokyo Chemical Society*, which is printed in Japanese, is quite closed to the scientific world.—I am, your obedient Servant,

JOJI SAKURAI.

The following letter was addressed by us to Professor F. W. Clarke on the subject of International Atomic Weights:—

Tokyo, Japan, March 28th, 1904.

Professor F. W. CLARKE, Chairman of the Sub-committee on International Atomic Weights.

DEAR SIR,—Whilst we wish to tender our sincere thanks to you and your worthy colleagues for the laborious task of yearly reviewing and compiling the table of atomic weights for international use, we feel at the same time bound to make some observations upon the course you have adopted in prosecuting the work entrusted to you.

The first and most important point is in reference to the introduction of the double standard. We can appreciate the delicate position which you occupy with respect to this much-controverted question, and we can also understand why you chose to leave the matter to take its own course without giving decision in one way or the other. But, as you must know, the larger committee on International Atomic Weights, from which the sub-committee has been formed, had decided, by a great majority of votes, to use *exclusively* those atomic weights which are based on the oxygen standard. The introduction of the double standard is therefore utterly against the above decision, and also in direct opposition to the very motive with which the International Atomic Weights Committee has been organised; moreover, the matter has thereby become worse than ever, for there are now two sets of atomic weights which are apparently equally authorised.

But, apart from the question of the previous history which the formation of the Sub-committee possesses, and which, as it appears to us, you have wholly disregarded, we beg to call your attention to the following circumstances:—

In looking over your tables of atomic weights for 1903 and 1904, we observe that, in the numbers under  $O = 16$ , only so many figures are given that the last figure may also be taken as correct. That you deem the adoption of this rule, which had been adhered to by the German Committee, desirable is evident from the remark you have made with respect to  $H = 1.008$  in your Report for 1903, but when we examine the atomic weights under  $H = 1$  we find curious contradictions. The atomic weight of oxygen is given with only four figures—that is,  $O = 15.88$ —from which we may conclude that you regard the ratio  $O : H$  has been only so far determined with certainty, and not further, and we are consequently at a loss to understand how the atomic weights of iodine, lead, and silver have been evaluated to five figures, as given. Professor Morley's ratio,  $O : H = 15.879$ , has apparently been employed for the purpose; but then, in accordance with the principle which

made you alter  $H = 1.01$  to  $H = 1.008$ , the atomic weight of oxygen should stand as  $O = 15.879$ , and not  $O = 15.88$ , as in your tables. This, it appears to us, illustrates in the most conspicuous manner the shortcomings of the hydrogen standard, which have already been pointed out by various chemists.

Again, on going through your tables, one is tempted to entertain the absurd notion that the atomic weights according to the hydrogen standard could be ascertained with greater accuracy than those based on the oxygen standard, for there are as many as eighteen elements whose atomic weights have more figures under  $H = 1$  than under  $O = 16$ , while there is only one element treated in the opposite manner. Of these eighteen elements thus favourably (?) treated under  $H = 1$ , the majority are rare elements, whose atomic weights are avowedly less accurate than the others. What is more remarkable still is the fact that one finds phosphorus among the number—an element whose atomic weight, according to your Report, "has been peculiarly neglected." It is clear that you do not have much confidence even in the value  $P = 31.0$  ( $O = 16$ ). What sort of reliance can, then, be placed in the last figure of the value  $P = 30.77$  given under  $H = 1$ ? Are these contradictions mere consequences of re-calculation of the atomic weights under  $H = 1$  from those under  $O = 16$ ? Or are there some other reasons for the adoption of these values?

However it might have been, the lack of uniformity in the two series is lamentable. This is only one of the unhappy consequences of retaining both standards. It might perhaps be urged that the uniformity in the treatment of figures can be ensured by taking ordinary critical care in re-calculation; but the fact that such re-calculation, which, moreover, involves the uncertainty of the ratio  $O : H$ , is at all necessary, is a great drawback of the hydrogen standard.

As Professor Ostwald strongly protested against the adoption of the hydrogen standard directly after the appearance of your first Report, we have been waiting for the publication of your second Report in the hope of seeing it dropped. But our hopes have been denied, and we feel ourselves obliged to address this letter to you.

There is another matter which, though of less importance, cannot be allowed to pass unnoticed, namely, the alteration of the symbols Be for beryllium and Nb for niobium into Gl (glucium) and Cb (columbium) respectively, which has been introduced into your tables. That atomic symbols ought to be as much international as atomic weights has been well recognised in the use, for example, of the symbol K for potassium. There is nothing which prevents the use of one name for an element in one country and another name in another, but surely atomic symbols should not be allowed to suffer similar discrepancies. The symbols Be and Nb have been and are most generally employed all over the world; if Gl and Cb had any claim to be considered as candidates, the choice ought to have been made according to the broadest international usage.

Lastly, we would like to call your attention to the unsatisfactory state of the relation which exists between the originally formed larger committee on International Atomic Weights and the Sub-committee of which you are the worthy Chairman—or, rather, non-existence apparently of any relation whatever between the two. Members of the larger committee do not receive any report from you, nor are they told through what periodicals your report will be made known to them. The matter of history has, it appears to us, been again disregarded here.

Trusting that the points represented above may receive your due consideration, and also that we may be allowed to publish this letter,—We are, Dear Sir, your obedient Servants,

K. IKEDA,  
JOJI SAKURAI,

Members of the Committee  
on International Atomic  
Weights, representing the  
Tokyo Chemical Society.

\* From the *Journal of the Tokyo Chemical Society*, April, 1904.

## PROCEEDINGS OF SOCIETIES.

## PHYSICAL SOCIETY.

*Ordinary Meeting, June 10th, 1904.*

Dr. R. T. GLAZEBROOK, President, in the Chair.

PROF. H. L. CALLENDAR gave a demonstration of the "*Projection of the Indicator Diagrams of a Petrol Motor.*"

The lantern-slides illustrated the working of the motor under various conditions, and were prepared to elucidate the nature of some of the defects which occur in practice. The motor itself was exhibited in action, with the indicator attached, and the actual diagrams were projected on the screen showing the changes of form as they occur when the conditions of running are changed. The motor employed was a Clement-Garrard cycle-motor, with 60 m.m. bore and 70 m.m. stroke. The inlet valve is automatic, and is provided with an arrangement for varying the tension of the spring for experimental purposes when the engine is running. The indicator used was a Hospitalier-Carpentier manograph, with certain modifications which are necessary for high-speed work. It is connected directly to the cylinder by a short steel tube in place of the long fine copper tube usually employed. The pressure acts on a thin steel disc, the movements of which are communicated to a small pivoted mirror, which is tilted upwards through an angle proportional to the pressure acting on the disc. The movement of the mirror is observed or recorded by a ray of light reflected on to a screen or photographic plate. In order to record the pressure at each point of the stroke in the form of a continuous curve or diagram, the mirror is simultaneously tilted from side to side by a little crank revolving in time with the fly-wheel, so that the spot of light reflected from the mirror moves horizontally in time with the motion of the piston in the cylinder. Owing to the high speed the spot of light is seen as a continuous curve, which is readily measured or photographed. The engine works on the four-stroke cycle of operations—suction, compression, explosion, and exhaust—and runs at speeds varying from 2000 to 2500 revolutions per minute.

Prof. Callendar exhibited a series of lantern-slides showing how the characteristics of the diagram depend upon the point of the stroke at which the gases are ignited. He also demonstrated experimentally how the pressure retardation, which occurs when using a long fine copper tube to connect a high-frequency engine to a manograph, could be compensated by retarding the position of the spot of light relative to the piston by an amount equal to the retardation of the pressure at any given speed. Leakage is a fruitful source of loss of power, but it was shown that it could easily be detected by an examination of the indicator diagrams. A leak shows itself at a loop at the end of the compression-stroke if the spark is retarded, coupled with a rapid fall of pressure during expansion. The effect of overheating of the engine can be well shown by comparing two diagrams taken under similar conditions with the cylinder cold in one case, but hot in the other. The hot cylinder shows the higher compression-pressure and the smaller explosion-pressure, so that the power is diminished for both reasons.

Diagrams were shown to illustrate the effects of back pressure, due to insufficient exhaust lift or to a throttled silencer. The loss of power on this account is not very serious unless the throttling is excessive.

Prof. J. A. FLEMING read a paper entitled "*A Model Illustrating the Propagation of an Alternating Current along a Telephone Cable, and a Simple Theory of the same.*"

Although the mathematical theory of the propagation of alternating currents along lineal conductors having capacity, inductance, resistance, and leakage has been developed by many writers in great fulness, the conclusions

reached by them have not always been readily assimilated by practical engineers, and in some cases unsound theories have been put forward regarding the conditions limiting telephonic speech along wires. The present paper contains an account of a model (exhibited at the meeting) which has been constructed by the author, for the purpose of explaining in a simple manner the physical meaning of the mathematical expressions which are reached in discussing the propagation of alternating currents along a telephone or telegraph cable. The first part of the paper is concerned with a presentation of the elementary theory of the phenomena expressed in terms of complex quantities, which forms a more suitable mode of investigation than the use of ordinary trigonometrical analysis, as the time-element is thereby eliminated. Beginning with the fundamental differential equations for the distribution of potential and current in an element of such a cable when traversed by any current, these are then thrown into the form appropriate when considering simple harmonic currents, and a solution given for the potential and current distribution in the case of a semi-infinite cable, having a simple periodic E.M.F. applied at the end. This solution is of the form  $V = Ee^{-\alpha x}(\cos \beta x + i \sin \beta x)$ , and it indicates that the potential,  $V$ , at any point in the semi-infinite cable has a maximum value which is less than that at the origin, ( $E$ ), in an assigned ratio, and shifted backwards by an assigned angle in phase. A geometrical interpretation was then given of the above equations, leading to a graphic representation of the distribution of potential in the cable at any moment, and the model shown illustrated the meaning of the above equation and the graphic interpretation of it. The model consists of a steel axis, on which are keyed a number of wooden discs grooved on the edge. These are set on the shaft eccentrically, and the eccentricity of the wheels diminishes in geometrical progression. Each eccentric is shifted backwards in phase compared with its preceding neighbour by an equal angle, and these eccentricities and phase-angles can be varied. Each eccentric wheel is embraced by a long string, the ends of which are attached to metal blocks which can slide up and down on a steel wire. These strings are all of equal length. When the axis carrying the wheels revolves, each block moves up and down with a nearly simple harmonic motion, but the amplitude of that motion decreases from block to block, and, moreover, the phase of the motion of each block is delayed behind that of its next preceding neighbour by an equal amount. Hence, when the axis is turned round, the blocks, of which there are 48, are arranged in a periodic curve of decreasing amplitude, and move up and down in a manner which imitates the propagation of a wave of alternating current or potential along a cable. The paper then proceeds with a discussion of the theory, and a very simple proof is given of Mr. Heaviside's condition for the distortionless propagation of a wave of electric current. The criterion of distortion is then applied in the case of one long submarine cable, and it is shown how far removed such a cable is from being a distortionless cable. The remedies which have been proposed are then discussed, and it is shown that it is hopeless to expect any real advantage merely by bringing the iron armour closer to the cable-conductor, but that a large reward awaits any inventor who can bestow upon a long submarine cable inductance up to 300 or 400 henries per naut, without at the same time increasing much the usual capacity of 0.3 m.f.s. per knot. Allusion is made to Prof. Pupin's investigations concerning the insertion of inductance-coils at intervals along the telephone lines, and the suggestion is made that there is no difficulty in applying this plan to subterranean telephone trunk-lines, and that considerable improvement in the transmission of telephonic speech could thereby probably be effected.

Mr. T. H. BLAKESLEY congratulated the author upon the interesting result contained in his paper, that under a certain critical relationship, among the properties of the cable relatively distortionless waves might take place whatever the frequency. But he thought some other of the

consequences of this relationship might be more simply exhibited in the forms for potential and current,—

$$V = Ee^{-\alpha x} \cos(\beta x - pt), \quad I = \frac{Ee^{-\alpha x}}{q} \cos(\beta x - pt + m)$$

where—

$$q^2 = \frac{R}{K} \left\{ \frac{1 + \left(\frac{Lp}{R}\right)^2}{1 + \left(\frac{Cp}{K}\right)^2} \right\} \quad \text{and} \quad \tan 2m = \frac{(LK - RC)p}{RK + LCp^2}.$$

$q$  is of the nature of resistance, and  $m$  is the angle of lag of the current behind the potential, and is constant for all points of the cable. This lag-angle disappears when  $LK - RC = 0$ , but from the expression for  $q^2$  it is clear that this quantity will be independent of the frequency if  $L/R = C/K$ , which is again the same condition. Thus the real degradation of current would be the same for all tones, and the conjunction of current and potential, for all points, would be secured by this condition. It would not, however, be easy in practice to fulfil this condition. There was, however, something to be said for keeping both  $L$  and  $K$  small, as in fact they are in practice. For, looking at the expression for  $q^2$ , it will be seen that it diminishes as the frequency rises if  $CR > LK$ , and this is true of cables in practice. Hence, though  $e^{-\alpha x}$  grows less with rising frequency the resistance  $q$  also becomes less, so that the degradation of current is not so great as that implied in the mere increase of  $\alpha$  with frequency. Mr. Blakesley pointed out that in operating upon equations 13 and 14 to obtain equations 20 and 21, the quantity  $\sqrt{(R^2 + p^2 L^2)(K^2 + p^2 C^2)}$  could be written in two ways:— $\sqrt{(KR \pm p^2 LC)^2 + p^2 (LK \mp CR)^2}$ . The author had chosen the top signs, but interesting results could be obtained by choosing the bottom signs and contemplating the equality of  $KR$  and  $p^2 LC$ .

The CHAIRMAN read a letter from Mr. Jacob pointing out that the dielectric resistance of the 1880 Anglo-American Cable, considered in the paper, is much higher, as it lies in the sea, than the figure quoted by the author, which was obtained at 75° F. and atmospheric pressure after 1 min. electrification. The electrification or absorption of the dielectric, however, masked all the effects of the high insulation value. An inductance of 300 or 400 henries per naut was beyond possibility; the inductance should not be of a higher order than one henry per naut.

Dr. FLEMING, in reply, said that the relation between the electrical constants of the cable which gave distortionless transmission was discovered and announced twenty years or more ago by Mr. Oliver Heaviside. All that he (Dr. Fleming) had attempted to do was to simplify and illustrate the proofs of this condition. The employment of complex quantities in the discussion of alternating-current problems led to a considerable simplification of the analysis. As in practice we were only concerned with the maximum or root-mean-square values of the potential and current, the introduction of the time-variable into the equations led to complicated expressions, the physical meaning of which was not readily apparent to most students. All the usual algebraic expressions for current and potential at any point could, however, be readily obtained from the vector equations he had given. As regards the practical realisation of Heaviside's distortionless conditions for submarine cables, the figures given in the paper were merely intended to indicate the remoteness of its possibility. The correction furnished by Mr. Jacob for the insulation resistance of the dielectric when the cable was immersed did not affect the general conclusions, as if the insulation resistance was greater, the departure from the distortionless conditions would be then still greater. He recognised that the "absorption" of the dielectric introduced a factor not taken into account in the mathematical theory. As regards the practical superior limit of inductance which might be given to a cable, no one could say what invention might not bring forth. He was glad

to hear that an inductance of 1 henry per naut might improve matters sensibly, because he had devised a plan by which an approximation to this might be reached. Mr. Jacob only confirmed the statement in his paper in expressing the opinion that an inductance of 300 to 400 henries per naut was impracticable.

Mr. M. E. J. GHEURY then exhibited a Gyroscopic Collimator. The instrument is used in connection with an ordinary sextant, the observation being taken as with the sea horizon by bringing the image of an observed body into a field of vision, in which a horizontal grating of a special kind allows the observer to ascertain the direction of the true horizon. The essential feature of the collimator is a gyrostat equivalent to a long pendulum, the period of vibration of which is very much greater than the periods of the various motions of the ship upon which it is used.

## THE FARADAY SOCIETY.

AN Ordinary Meeting of the Faraday Society was held on Thursday, June 9th, at the Institution of Electrical Engineers. The President, Dr. J. W. SWAN, was in the Chair.

Mr. ADOLPHE MINET presented the first part of a paper on "*The Electric Furnace, its Origin, Transformations, and Applications.*"

The paper discusses the growth of the furnace from the historical point of view, and then proposes a new classification, which is worked out in minute detail in the form of a table. A full bibliography of the electric furnace completes this section of the paper.

This discussion was postponed until the autumn, when the author will complete his study and fully illustrate his subject.

Dr. F. M. PERKIN described a form of Porous Diaphragm that he has found convenient for laboratory use. It consists of two perforated concentric porcelain cylinders packed in between with brown paper, asbestos, or other material, depending on the use to which the diaphragm is to be put.

Mr. G. T. BEILBY read a paper on "*The Hard and Soft States in Metals.*"

The wide range of the phenomena which are directly or indirectly associated with the hard and soft states in metals indicates that a knowledge of these states is of fundamental importance. An exclusively crystalline theory of metal structure, even when stretched to its widest limits is insufficient fully to explain these phenomena. But the crystalline is not the only form of solid aggregate; the movement of the molecules in the liquid state may be so suddenly arrested that they have no time to fall into the regular formation of the crystalline state, so that the solid which results is amorphous, not crystalline. A suddenly congealed liquid may be likened to an instantaneous photograph of the rapidly moving molecules of the liquid state.

The views here advanced are based on the author's earlier observations on surface flow in crystalline solids. The evidence afforded by the micro-structure has been supplemented by observations on the other properties of metals in the hard and soft states, and the view is now advanced that these states are perfectly distinct phases. This is shown by the mechanical, electrical, optical, and thermo-chemical properties as well as by the micro-structure. The soft phase, C, is crystalline, and the hard phase, A, is amorphous.

The transformation of A into C is effected by heat, and takes place at a definite transition-temperature. On either side of the transition-point the various properties are characteristic and distinct; for instance, an E.M.F. of 120 micro-volts may be developed in a thermo-junction of silver in the two phases. The E.M.F. falls to zero after the junction has been heated to 260° for a few seconds. Silver

leaf, which is opaque and highly reflecting below the transition temperature, becomes transparent and very much less reflecting if kept for a time at a temperature a little above that point.

The transformation from hard to soft is thermally irreversible, but is readily effected mechanically. This reverse transformation—soft to hard—takes place when the metal is deformed by over-strain, however slight. It takes place through an intermediate mobile phase in which the molecules have a freedom analogous to that in the liquid phase. This freedom is produced by motion directly imparted to the molecules during the slipping of one portion of the material over another. The state of the mobile phase is somewhat analogous to that of an undercooled liquid. This transformation, C—M—A, while it occurs at every moving surface, does so, as a rule, in extremely thin layers. The layers of the hard phase which result supply a rigid casing for the unaltered crystalline elements, and thereby give a granular and cellular structure to metal which has been overstrained by having any kind of work done on it. The co-existence of the two phases in this way accounts for the variety of structure which may be developed in malleable and ductile metals.

Dr. T. M. LOWRY said that the temperature which Mr. Beilby called the transition-point was not a true *transition-point*, such as exists, for example, in sulphur at  $96^{\circ}\text{C}$ ., at which the rhombic passes into the monoclinic phase, but really the *stability-limit*; that is, the temperature at which the mobility of the molecules enables a change which has been trying to take place all along to do so at a visible velocity.

Mr. J. C. M. GARNETT suggested that the different results obtained in the two methods—by transmitted and reflected light—of measuring the optical constants might be explained on Mr. Beilby's hypothesis of an A and C phase.

Dr. C. M. DESCH thought that the existence of a thin transparent film on the surfaces of polished metals might explain the Kerr effect.

#### THE ROYAL SOCIETY.

##### CONVERSAZIONE AT BURLINGTON HOUSE.

ON Wednesday evening last, June 22nd, a most successful Conversazione was held at the Royal Society's Rooms at Burlington House, the occasion being known as "Ladies Night," and the guests were received by Sir William and Lady Huggins.

A certain number of the exhibits were shown at the last Soirée, on May 13, and have been already noticed in these columns.

Among the most interesting of those to be seen on Wednesday evening last we may mention a very ingenious instrument, exhibited by Mr. W. DUDDALL, called the Thermo-galvanometer, an instrument for measuring very small alternating currents. The instrument is intended for the measurement of very small rapidly-varying currents, such as telephonic currents and the currents produced in the receiving vertical wire in wireless telegraphy. Signals were sent from a transmitter at the end of the main library (Room V.); the very small current produced in the vertical wire at the receiving station was measured with the thermo-galvanometer, and the great increase in the current due to tuning the receiving circuit was demonstrated. Some experiments with telephonic currents were shown. The sensibility of the instrument is such that either direct or alternating currents from a few micro-amperes upwards can be measured. The deflection is practically proportional to the mean squared value of the current flowing.

Mr. W. HIBBERT showed a new Magnetic Balance, constructed as follows:—The beam of a balance is made of a magnetised steel rod 27 c.m. long. The "centres" of the poles are 25 c.m. apart. The repellent pole of a second

magnet being placed over one end of the beam causes this to descend, and the force of repulsion is balanced by a weight sliding on the other half of the beam. The absolute value of a pole in C.G.S. units can be ascertained in a very few minutes, without reference to the terrestrial or other field. The instrument can also be used for finding the approximate value of an electric current.

A simple method for printing without the use of inks was shown by Mr. FRANCIS SHERIDAN called the Physiotype. It is specially adapted to finger printing and the reproduction of designs from animal and vegetable life. The subject to be reproduced is pressed on paper, and an invisible impression is developed with a coloured powder into a dark and permanent print.

The Cilioscribe, exhibited by Dr. W. E. DIXON and Mr. O. INCHLEY, is a machine to record the movements of cilia and the effect of physical conditions and chemical reagents upon them. The apparatus consists of an upright silver rod free to revolve on its vertical axis. A light aluminium wheel is fixed to the upper end of this and a short ebonite spindle a few centimetres lower, all having the same vertical axis. The ciliated mucous membrane from the frog's pharynx is wrapped round the spindle. Hooks are fixed into the ends of the membrane and connected to a silk thread which passes over a pulley to a weight. The cilia turn the spindle, and a record of their activity is obtained by a time marker recording seconds on a blackened paper attached to the aluminium wheel.

Mr. C. E. S. PHILLIPS showed an Automatic Vacuum Pump of novel construction. The apparatus consists of a modified Toepler pump, so arranged that it works automatically through the operation of electrically controlled devices, for the purpose of producing extremely high rarefactions. The pump will reduce the gas pressure within a vessel of 200 c.c. capacity from that of the atmosphere to  $0.002\text{ m.m.}$  in fifteen minutes. By the action of the mercury in completing the circuit of a relay a solenoid is excited which operates a four-way slide valve, and this, in its turn, connects the lower chamber of the Toepler pump with either the atmosphere or a mechanical pump, depending upon whether the mercury touches the lower contacts or those in the air trap on the left hand side of the apparatus. Particular attention is drawn to the adjustable device for causing the mercury to pause before ascending into the upper chamber.

Mrs. AYRTON showed some striking experiments on the Origin and Growth of Ripple Marks. The experiments illustrate the way in which the sand ripples are formed on the sea shore. If sand be spread quite evenly on the bottom of a trough, and water above the sand be oscillated so as to produce stationary waves, a small ridge is formed where the horizontal velocity of the water is greatest, next a ridge is started on each side of the first, which grows; then two more ridges are started, the former growing, and so on until the whole surface of the sand is ripple-marked. Each ripple now slowly moves towards the place of greatest horizontal velocity, while fresh ripples form near the places of least horizontal velocity. Pairs of ripples then coalesce here and there, and finally the greater part of the sand is assembled in a ripple-marked heap at the places of greatest horizontal velocity, this final result being attained, for example, in about twenty-five minutes in the case of the six foot trough exhibited, when the stationary wave is twice the length of the trough. The vortices which are produced in the lee of each ridge during a part of each swing of the water are shown by means of pepper, and the way in which these vortices build up the ridges is exhibited.

During the evening the following demonstrations were given, with Lantern illustrations:—

Lantern Slides, illustrative of—(1) Operations at the Simplon Tunnel; (2) The Victoria Falls and Gorge of the River Zambesi, and proposed Bridge, by Mr. FRANCIS FOX.

The Charge of Negative Electricity fired off by Radium, by the Hon. R. J. STRUTT.

Colour Photographs of Living Insects, by Mr. F. ENOCK, F.L.S. This last lecture was of great interest. By no other means than that of colour photography would it be possible to reproduce the natural colours of living insects and their environment. The protective colouration and markings, as well as the natural pose of the insect, are all brought out in a marvellous manner. Insignificant markings, when seen from different points of view, are shown to be of the highest importance for the protection of the insect.

## NOTICES OF BOOKS

*Radio-activity.* By E. RUTHERFORD, D.Sc., F.R.S., F.R.S.C., Macdonald Professor of Physics, McGill University, Montreal. London; C. J. Clay and Sons.

IN this book, dedicated to "J. J. Thompson as a tribute of respect and admiration," the author has endeavoured to give a complete account from a physical standpoint of the properties possessed by the naturally radio-active bodies. As our knowledge of radio-activity has been obtained chiefly from information gained by research into the electric properties of gases, this latter subject is rather fully discussed, and a valuable chapter is included giving the various forms of apparatus used, and the methods for the measurement of radio-activity generally in use. The book constitutes a complete summary of the work that has been done upon this very interesting subject, and although some readers may not be prepared to accept at once all the speculations and hypotheses set forth, it is both interesting and valuable to have a clear statement of the grounds upon which have been based some of the most startling conclusions of recent times.

The ionisation theory of gases and the part it has played in furnishing a satisfactory explanation of the passage of electricity through vacuum tubes is discussed in Chapter II., where also the measurement of radio-activity by the determination of the "saturation current" is fully described, as are also C. T. R. Wilson's experiments on the condensation of water upon nuclei, and the use they have been put to by Prof. J. J. Thompson in his recent researches, forming as they do links supporting the series of interesting speculations that are now woven round the phenomena of radio-activity.

In Chapter III., "Methods of Measurement," the action of the rays upon a photographic plate, though valuable as a means of investigating the curvature of the path of the rays where deflected in a magnetic field and similar phenomena, is open to many restrictions as a means of general investigation, and the electric methods for the measurement of the "saturation current" is to be preferred. Various forms of electroscopes are described; one very useful form used by M. and Mme. Curie in their earlier researches is illustrated in full; with this instrument comparative tests of the radio-activity of various materials can be rapidly made, but it is not suited for absolute measurement. A modified form of electroscope has recently been devised by Mr. C. T. R. Wilson that is likely to be very useful. It consists of a small rectangular brass box containing at one of its ends a brass plate insulated and maintained at a constant potential of 200 volts; through the opposite side of the box a wire carrying a very narrow gold-leaf is fixed, insulated by a sulphur bead; normally the gold-leaf is attracted to the charged plate, and the box is tilted to the angle of maximum sensitiveness, about 30°. The other end of the wire carrying the gold-leaf is connected to the insulated system whose rise or fall of potential is to be measured; a movement of 200 scale-divisions was obtained by a rise in potential of only one volt above the case.

The construction and method of using electrometers is very thoroughly set out, and the chapter concludes with a description of M. Curie's quartz piezo-electric, an instru-

ment little known on this side of the Channel, but one that is particularly adapted to the measurement of saturation currents.

Chapter IV. plunges boldly into the nature of the radiations;  $\alpha$ -,  $\beta$ -, and  $\gamma$ -rays are each discussed separately in great detail; a description is given of Mr. Strutt's ingenious apparatus for demonstrating that a radium compound acquires a positive charge if contained in an envelope sufficiently thick to absorb the  $\alpha$ -rays, but thin enough to allow most of the  $\beta$ -rays to escape, and also an account of Sir William Crookes's spintharoscope for rendering visible the impacts of the  $\alpha$ -rays.

Chapters V. and VI., deal with the rate of emission of energy and the properties of radiation; the effect of radium in causing a spark to pass an alternative spark gap and its effect upon selenium are mentioned, as are also its action upon liquids and some of its chemical properties. Several chapters are devoted to the fundamental experiments that make the foundation of the disintegration theory. It is well that the author's views on this important subject have been clearly set out, and this section of the work deserves very careful study by any who may have been startled by the boldness of the views recently propagated. The physiological action of radium rays and their action upon certain microbes is of interest, being fraught with great possibilities. Sir William and Lady Huggins made the discovery that the light from a phosphorescent radium compound in air shows the line spectrum of nitrogen which can be photographed if sufficiently long exposure is given; this shows that radium at ordinary temperatures is able to set up radiations that are otherwise only produced by the electrical discharge under special conditions. Chapter X. deals with the law of decay and recovery of radio-activity in emanating substances; the theory of radio-active change is elaborated on pages 323-331, including the author's diagrams illustrating the course of alteration in uranium, thorium, and radium, and a full account is given of the experiments of Prof. Sir. W. Ramsay and Mr. Soddy upon the production of helium from radium and radium emanation.

The book may be truly said to contain all that is known about the radio-active elements, as well as setting out clearly the very interesting though perhaps revolutionary theories that have been devised to account for their remarkable properties, and it is likely for some time to come to be regarded as the standard work upon the subject.

*The Extra Pharmacopœia of Martindale and Westcott.* Revised by W. HARRISON MARTINDALE, Ph.D., F.C.S., and W. WYNN WESTCOTT, M.B., D.P.H. Eleventh Edition. London: H. K. Lewis. 1904. Pp. 809.

SINCE the publication of the tenth edition of the "Extra Pharmacopœia" three years ago, William Martindale, the originator of the book, has passed away, and the revision of the work was entrusted to his son, W. Harrison Martindale, and Dr. W. Wynn Westcott.

In this edition many of the older drugs, chemicals, and preparations have been omitted, thus enabling the revisors to add more than three hundred new remedies, drugs, &c. The size and weight of the volume have been decreased, though it contains 112 pages more than the last edition, by using finer paper, without impairing the clearness of the type.

One important addition is the section on Radiology, in which are introduced for the first time notes on radium, Röntgen rays, high-frequency currents, the Finsen lamp, and radiant heat in their important applications to therapeutics. Similar fresh monographs appear on amygdala, colchicum, lecitithin, ptomaines, &c., and the section on Mineral Waters contains information of about 150 mineral waters in various parts of the world. The water analysis notes and bacteriological notes have been revised and corrected up to date, and notes on the examination of stomach contents appear for the first time. The index has been revised and re-arranged, internal remedies being

printed in Roman type. while those for local or external use are in italics.

*Subject-list of Works on Electricity, Magnetism, and Electro-technics in the Library of the Patent Office.* London: Darling and Son, Ltd. 1904. Pp. 286.

THIS subject-list is No. 14 of the Patent Office Library series, and comprises 2374 works, representing 3792 volumes. The catalogue entries relating to these works number 2918, and are distributed under 307 headings and sub-headings. As an example of the system adopted, if a searcher requires information on Electro-plating of gold and silver, for example, the subject of Electro-plating will be found in the general alphabet under that heading. Further information will be obtained by referring to the key in which the headings are shown in class order. Here he will find the headings "Electro-plating," "Electro-deposition," "Electro-chemistry, Technology of," &c., which should next be consulted.

*Immune Sera; Hæmolysins, Cytotoxins, and Precipitins.* By Prof. A. WASSERMANN, M.D. Authorised Translation by CHARLES BOLDUAN, M.D. First Edition. London: Chapman and Hall, Limited. New York: John Wiley and Sons. 1904. Pp. 77.

THE subject of immunity and the reactions which take place when the blood of one animal is injected into the body of another are matters of interest, and are now becoming also of great importance.

At first these experiments, like so many others, were of scientific interest only, but they have been followed up and observed with such minute care that the results have been found applicable to many clinical questions, as well as to certain other problems of every-day life.

In this book we find described in the clearest manner the results of a large number of experiments by which the author hopes to introduce to his readers the essentials of the subject.

The bulk of the experiments herein described have to do with the injection of the blood or serum of one animal into the body of another, and the first important fact noticed in this connection was the following:—That if horses were injected with red blood cells of rabbits, the serum thereafter obtained from the horses would have acquired an appreciable toxicity for rabbits, and it was found further that this toxic action was really a dissolving power acquired by the horse serum for the red corpuscles of the rabbit. From these early observations the author and others have built up what is practically a new branch of biological science, and which is, moreover, leading up to important results bearing on artificial immunity from infectious diseases.

After it had been found that injection of an animal with red blood cells of another animal was followed by the production of definite specific reaction substances, investigators experimented to see whether this was also the case if other animal cells were used, and it was found that a series of reaction substances resulted, entirely analogous to the hæmolysins obtained from the red blood cells; these sera have been called cytotoxins, and are specific for the cells used for injection.

Another class of bodies are called precipitins, and they develop in the serum by treating an animal with albuminous bodies of another animal, and precipitate these albumins when the sera of the two animals are mixed. This power has been found to be very extensive, but it is not entirely specific; for instance, the serum of rabbits injected with chicken serum is a precipitin, not only for chicken serum but also for that of pigeons; while the serum of rabbits treated with that of guinea-pigs is a precipitin also for the serum of monkeys. The author, however, has elaborated a method for distinguishing the albumins from one another, and particularly to distinguish those derived from man from those of other animals. The reaction is

specific except that the serum of an animal treated with human serum reacts also with the serum of monkeys, but not with that of any other animals so far investigated. The book is one of great interest, and we recommend it cordially.

## OBITUARY.

THOMAS BAYLEY.

WE regret to announce the death, on June 7th last, from cardiac failure, of Mr. Thomas Bayley.

Mr. Bayley was the compiler of the well-known "Chemists' Pocket-book," by which work he was best known. He was, however, devoted to original research, which he pursued with unflagging ardour in such time as was available to him from his work as a manufacturing and consulting chemist. His work lay chiefly in the domain of the Periodic Law, and he published several papers in the *CHEMICAL NEWS*, *Journal of the Chemical Society*, *Philosophical Magazine*, and *Journal of the American Chemical Society*. Several new and important relations were laid bare in the course of these investigations, especially one deduced graphically from the At. Vol.-At. Wt. diagram. The result is a power of really accurate prediction of the properties of unknown elements, which has been verified in several important cases.

Mr. Bayley was also the author of "A Pocket-book for Pharmacists," and of "The Assay and Analysis of Iron and Steel, Iron Ores, and Fuel"—a work now out of print, but still in demand.

H. S. H.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise express'd.

*Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences.* Vol. cxxxviii., No. 21, May 24, 1904.

$\gamma$ -Diphenylanthracene and Symmetric  $\gamma$ -Diphenylanthracene Dihydride.—A. Haller and A. Guyot.—The authors prepare  $\gamma$ -diphenylanthracene in a pure state, and various derivatives of this body.

Direct Hydrogenation of Homologues of Aniline.—Paul Sabatier and J. B. Senderens.—For the amines of the first group (methyl- or ethylanilines) hydrogenation can be effected by means of nickel between 160° and 180°, and takes place quite regularly. The production of condensed amines, which takes place in the case of aniline, is impossible for the tertiary amines. The only secondary reaction being the production of a little formenio amine and benzene, transformed itself into cyclohexane. Ethylaniline,  $C_6H_5NHC_2H_5$ , gives cyclohexylethylamine,  $C_6H_{11}NHC_2H_5$ ; Diethylaniline,  $C_6H_5N(C_2H_5)_2$ , giving cyclohexyldiethylamine,  $C_6H_{11}N(C_2H_5)_2$ ; and dimethylaniline,  $C_6H_5N(CH_3)_2$ , giving cyclohexyldimethylaniline,  $C_6H_{11}N(CH_3)_2$ .

Thermic Ionisation of Saline Vapours.—G. Moreau.—When saline vapours are raised to a high temperature—say, 1000°—they become conductors, and remain conducting down to ordinary temperatures. The conductivity depends on the nature of the salt and its chemical constitution. The author's experiments on this subject show—(1) For a solution of constant concentration of a salt, A, the current,  $i$ , at first proportional to the voltage of the current, tends towards a limit,  $I$ , when this increases; (2) for the same salt the current limit  $I$  is proportional to

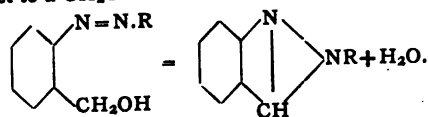
the concentration of the solution if this is very dilute (1 gm. per 1000), and varies more slowly; it tends towards a limit, J, attained more rapidly the less conducting the vapour; (3) of all the salts examined, those of potassium are more conducting than others, and their conductivity limit, I, depends on the radical acid; KI, KCl, KBr, and  $\text{KNO}_3$  are three or four times more conducting than the other potassium salts; (4) for the same salt, A, the conductivity is only apparent if the porcelain tube is incandescent, and this increases rapidly with the temperature.

**Cryoscopic Examination of Solutions in Antimony Sulphide.**—MM. Guinchant and Chrétien.—The authors investigate the subject of cryoscopy at very high temperatures. By the use of thermo-electric couples, the temperature of the medium can be accurately determined. The experiments were made with silver sulphide, values being obtained for the weight of this sulphide dissolved in 100 grms. of antimony sulphide, and for the lowering of the solidification-point of antimony sulphide in consequence. Experiments were also made with lead, nickel, and antimony sulphide.

**Estimation of Atmospheric Formaldehyde.**—H. Henriet.—The author describes the experiment by which he estimates the amount of formaldehyde in atmospheric air. The proportion he ascertains to be from 1/100,000 to 5/100,000 of the weight of the air.

**Method of Identifying the Fatty Acids.**—René Locquin.—M. Bouveault a short time ago described a method of identifying alcohols by preparing their pyruvic ethers and combining these with semicarbazide. Crystalline compounds are thus easily obtained. The author evolves a similar method of identifying the fatty acids. All these acids form with acetol or oxyacetone,  $\text{CH}_3\text{—CO—CH}_2\text{OH}$ , ketonic ethers susceptible of giving also semicarbazones, which constitute the characteristic derivatives of the acids to which they correspond. He proves that the semicarbazones thus obtained easily crystallise, are easy to purify, and have very defined fusing-points.

**Transformation of Azoic Compounds with Ortho-substituted Alcohol Function into Indazylic Derivatives.**—P. Freundler.—The author showed that benzene-*o*-benzylic alcohol yields phenylindazol when it is distilled or heated at  $100^\circ$  with dilute sulphuric acid. This reaction is absolutely general, and is effected each time that the azoic group is situated in the ortho-position with respect to a  $\text{CH}_2\text{OH}$  function:—



**Limit of the Union of Diazobenzene and Phenol.**—Leo Vignon.—By the direct action of one molecule of diazobenzene chloride on one molecule of phenol the author obtains paraoxyazobenzene. Two molecules of diazobenzene chloride acting on one molecule of phenol give an almost theoretical yield of phenolbisdiazobenzene. The formation of this latter body represents the limit of the union. In no case has phenoltridiazobenzene been obtained.

## MISCELLANEOUS.

**Action of Peroxide of Nitrogen on the Isonitroso-malonic Ethers.**—L. Bouveault and A. Wahl.—If peroxide of nitrogen in excess is made to act on 50 grms. of isonitroso-malonate of ethyl cooled in ice, at first the peroxide is dissolved, with the production of a brownish green colouration. In a short time bubbles of gas are formed throughout the mass. The temperature must be kept down, and after an hour or two the reaction is complete

and the liquid is decolorised. The product is then submitted to fractional distillation *in vacuo*, and two principal fractions are obtained; one boiling at  $95\text{--}110^\circ$  under 12 to 15 m.m., the other at  $110\text{--}130^\circ$ . The aqueous solution of the first one gives crystals of hydrate of mesoxalate of ethyl. The second fraction is treated with ammoniacal water, and the yellow solution shaken up with ether, decanted, and decomposed with a dilute acid. The precipitated oil is purified and dissolved in absolute alcohol, and an excess of ammoniacal alcohol added. After some time a salt is deposited; this is re-crystallised in alcohol, when yellow flakes are formed, consisting of the ammoniacal salt of the nitromalonate of ethyl.—*Bull. Soc. Chim.*, Series 3, xxix., Nos. 18—19.

**Production of Pure Iodine, and the Preparation of Titrated Solutions of Iodine and Sodid Hyposulphite.**—L. de Koninck.—The author reviews the different known methods for procuring pure iodine for the preparation of titrated solutions. He recommends the action, by the dry way, of bichromate of potassium on iodide of potassium, feeling assured that the impurities usually contained in these two salts are generally without influence on the final product; further, it is very easy to purify these salts. The iodine is prepared by this method, by heating a very intimate mixture of one part of iodide with three-fourths of bichromate. By collecting the iodine in a tared matras or glass bulb, it is possible to prepare a titrated solution directly and rapidly.—*Bull. Assoc. Belge Chim.*, vol. xvii., p. 15.

**Electrolytic Estimation of Thallium (Anodic Precipitation of the Oxide).**—E. Haberg.—Excellent results can be obtained by electrolysis a sulphuric solution of thallium; 0.2 to 1 gm. of the sulphate are dissolved in a platinum crucible in 80 to 100 c.c. of water; add 2 to 6 c.c. of normal sulphuric acid and 5 to 10 c.c. of acetone, electrolyse with a current varying from 1.7 to 0.3 volts at the end of the operation. The platinum crucible serves as the anode; the cathode is formed of a disc of platinum. The intensity of the current should not be more than 0.02 to 0.05 ampères at the outside; the temperature should be maintained between  $50$  and  $55^\circ$ ; the volume of the liquid should remain constant. The precipitated oxide is washed in water, twice with alcohol, once with ether, and dried for twenty minutes at  $160\text{--}165^\circ$ . The addition of an alkaline sulphate facilitates the electrolysis by increasing the conductivity of the solution, but in this case we must carefully wash the precipitated oxide or else we should obtain too high a figure.—*Zeit. Anorg. Chem.*, vol. xxxv., p. 347.

**A New Method for the Estimation of Selenium.**—A. Gutbier and E. Rohn.—The authors have applied to the estimation of selenium, the method mentioned by one of them for the estimation of tellurium (*Zeit. Anorg. Chem.*, vol. xxxii., p. 295). The selenium should be in the form of selenious acid, for, contrary to what has been observed with tellurium, the aqueous solutions of selenic acid are only slowly and incompletely reduced by hypophosphorous acid. In the presence of concentrated hydrochloric acid we obtain only a small quantity on selenium dissolved in the colloidal state, without any precipitate being observed. The aqueous solutions of selenious acid are precipitated slowly in the cold, and rapidly when heated. The selenium obtained is quite comparable with the tellurium produced under the same conditions. By prolonging the heating the reduction goes as far as seleniuretted hydrogen. For the quantitative determination of selenium the operations should be carried out in alkaline solution. Instead of 71.19 of selenium the authors found 71.22, 70.99, 71.08, and 71.25.—*Zeit. Anorg. Chem.*, vol. xxxiv., p. 448.

**The Estimation of Iron in the presence of Zirconium, by Rivot's Method.**—K. Daniel and H. Leberle.—Rivot's method for the separation of two oxides of which one only is reducible by hydrogen, does not apply to the separation of oxide of iron and zirconium. The reduction of the iron only takes place in anything like a complete



manner in the presence of a very small quantity of  $ZrO_2$ . With 1 equivalent of Zr for 50 of iron, the figure found for the iron is 0.1822 instead of 0.1830. The error increases rapidly with the percentage of zirconium. In a mixture of equal parts of iron and zirconium, the authors found 0.0882 of iron instead of 0.0933. In the experiments of Gutbier and Hüller, who recently described this method (*Zeit. Anorg. Chem.*, vol. xxxii., p. 92), and appeared to have arrived at very concordant results, the figures found are a consequence of the incomplete drying of the oxides employed. The authors have, in fact, made certain that the oxides of iron and of zirconium dried at  $105^\circ$ , as in these last-named chemists' experiments, retain notable quantities of water. The loss of weight observed results, therefore, from (1) the actual reduction of a portion of  $Fe_2O_3$ , and (2), from the dehydration of the hydrated oxides ( $Fe_2O_3 + ZrO_2$ ).—*Zeit. Anorg. Chem.*, vol. xxxiv., p. 393.

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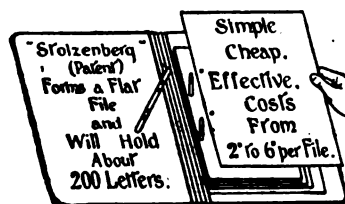
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## INDEX.

- ABNEY, Sir W. de W.**, calculation of colours for colour sensitometers, and the illumination of three-colour photographic transparencies by spectrum colours, 212
- Accumulator, electric**, 287
- Accumulators, electric**, regulations for manufacture, 35
- Acetaldehyde solution**, action of hydrogen sulphide on, 260
- Acetals, nitrobenzoic**, reduction, 119  
of polyatomic alcohols of the sugar series, 238
- Acetates of the alkaline earths**, 34
- Acetol, methylic ether of**, 251
- Acetone hydrates**, 244
- Acetylene gas**, application to heating of germination stoves, 215
- Acetylalcohol, nitration**, 287
- Acid, abietic**, constitution, 259
- allantoic**, 143
- benzoic, organo-metallic compounds of**, 83
- bromosuccinic**, action on the pyridic and quinolic bases, 34
- bismuthogallic**, constitution, 23
- boric**, 262  
and strong acids, estimation, 60
- bromosuccinic**, action on the pyridic and quinolic bases, 34
- $\alpha$ -campholenic racemic**, derivatives, 191
- $\alpha$ -campholytic**, derivatives, 191
- carbonic**, action on solutions of sodium nitrite, 191
- chaulmoogric**, constitution, 295
- chloric**, action of copper on, 269
- electrolysis of**, 106
- chlorocamphorsulphonic  $d$ - and  $d$ - and  $l$ -methylhydri- amines**, isomeric salts of, 19
- crotonic**, separation of  $\beta$ - from  $\alpha$ -, 140
- $\alpha$ - $\beta$ -dihydroxybutyric**, resolution into its optically active constituents, 93
- dilute**, apparent diminution of energy of in presence of a neuter salt of this acid, 250
- ethylhomocamporic**, 168
- gallic**, action of hydrated oxide of bismuth on acid isomers of, 227
- hydriodic**, influence on oxidation of sulphurous acid, 238
- hydrochloric**, and oxygen, action of a mixture of on metals, 33
- hydrocyanic**, action on ammonium aldehyde and analogous combinations, 22
- hydroxystearic**, 294
- hypophosphorous**, preparation and properties, 297
- isopyromucic**, ethers of, 22
- ketohexahydrobenzoic**, 141
- $l$ -mandelic**, esterification by menthol and borneol, 116
- methylsuccinic**, acid esters of, 177
- molybdic**, colour reactions of, 106
- $o$ -nitrobenzoylacetic**, 70
- oxalic**, and potassium permanganate, speed of reaction between, 96
- peroxylmagnesiumsulphonic**, 18
- phosphoric**, determination, 73, 89, 101
- etherification by glycerin**, 34
- salicylic, organo-mercuric compounds of**, 120
- Acid, silicic**, 251, 263  
and alkaline and alkaline-earthly silicates, 23
- sulphurous**, influence of hydriodic acid on oxidation, 238
- trimethylbutyric,  $\gamma$ ,  $\gamma'$ ,  $\gamma''$** , 226
- trimethylglutamic**, cis- and trans-modifications of, 80
- vanadic**, and ethenol, colour reactions of, 82
- Acids, acetylenic**, 226  
decomposition by the alkalis, 226  
hydration, 226
- benzoic**, diortho-substituted, 19
- caprylic and pelargonic**, synthetals, 226
- crotonic,  $\gamma$ -substituted**, 262
- $\alpha$ -dimethylglutaric and  $\alpha$ -dimethyladipic**, synthesis of, 168
- fatty**, identifying, 311
- solubility of salts of lower**, 193
- ferro- and ferri-hydrocyanic**, chemical equilibrium between, 199
- hydroxycarboxylic**, action of heat on, 81, 294
- $\beta$ -ketonic and  $\beta$ -aldehydic**, optically active esters of, 21
- manganous and tungstic**, complex double salt of, 288
- methodic degradation**, 191
- methyladipic,  $\beta$ - $\alpha$ -substituted**, 107
- methylcinnaemic,  $\beta$ -isomeric**, 251
- monoalcoholphosphoric**, silver and lead salts of, 203
- nitrobenzoic**, reduction, 119
- oxalcoyl-ethylenic**, 119
- pelargonic and caprylic**, synthetals, 226
- resin, of conifers**, 259
- strong**, and boric acid, estimation, 60
- Ackroyd, W.**, action of radium rays on halides of the alkali metals, 246  
principal cause of saltiness of the Dead Sea, 13
- Acridine series**, 70
- Acylaminoketones**, isomeric change of diacylanilides into, 117, 176
- Acyl thioyanates**, tautomeric character of, 92
- $\text{Ether}$** , absence of effects of motion through in relation to constitution of matter, 284
- "Africa, South, Proceedings of Chemical and Metallurgical Society of"** (review), 46
- Africa, South, Chemical, Metallurgical, and Mining Society of**, 71
- "Agricultural Work in the Botanic Gardens and the Government Laboratory at British Guiana, Report"** (review), 274
- Air, formic aldehyde in**, 23, 120
- Alcohol, campholenic**, and campholenic derivatives, 119
- hydrates**, ethylic, 22
- methylac**, 244
- isopropyl**, conversion into isopropyl ether, 260
- methyl**, estimation in presence of ethyl alcohol, 18
- methyllic**, electric osmotic in, 237, 286
- primary phenylethyllic**, 298
- trichlorisopropyllic**, 106
- Alcohols, acid, nitric ethers of**, 35, 59, 227
- Alcohols, amino**, of tertiary alcoholic function, 203  
boiling-points, 265  
condensation of acetylenic ethers with, 107
- hexatomic**, and mononitrobenzaldehydes, combination, 23
- hydro-aromatic**, preparation, 59
- polyatomic of sugar series**, acetals of, 238
- primary**, preparation by means of the corresponding amides, 94
- secondary and tertiary of fatty series**, differentiation, 287
- purification and identification**, 251
- tertiary**, application of Grignard's reaction to the halogen ethers of, 275
- synthesis**, 95
- Alcoyl and alcoylidenic derivatives of cyclic ketones**, preparation, 286
- Alcoyl-allyl-ketones**, 119
- Alcoyl menthones**, preparation, 286
- Aldehyde, formic**, in air, 23, 120
- $\beta$ -oxy- $\alpha$ -naphthoic**, derivatives and products of condensation, 287
- Aldehydes, aromatic**, synthesis, 83  
boiling-points, 265  
condensation with menthyl acetacetate, 21
- fatty**, synthesis, 226
- preparation**, 191
- reaction for**, 131, 251
- synthesis**, 83, 191
- transformation of primary  $\alpha$ -glycols** into the corresponding, 59
- Alkali earth metals, periodides of**, 273
- metals**, action of radium rays on halides of, 246
- Alkaline and alkaline-earthly chlorates**, electrolysis with copper anode, 293
- carbonates**, behaviour of phenolphthalein in presence of, 27
- dissociation**, 59
- chlorides**, electrolysis of resistance of iridio-platinum anodes used in, 138
- earth mangan-manganates**, 155
- metals**, fluochlorides, fluobromides, and fluoiodides of, 106
- periodides of**, 273
- earths, acetates of**, 34
- hydroxides and ammonia**, relative strengths of as measured by their action on cotarnine, 17
- metals**, flame spectra of, 130
- nitroprussiates**, estimation, 114
- Alkaloids**, precipitation by nitrate of uranium, 26
- Alkali iodides**, chemical dynamics of, 140
- Alkylsilicon chlorides**, preparation, 81
- Alkyl ureas**, decomposition, 273
- Allantoin**, 143
- Allen, E. T.**, precipitation and separation by weak organic bases, 43, 63, 76, 88, 103
- Allyl ketones**, 179
- Aloy, J.**, precipitation of some alkaloids by uranium nitrate, 26
- Alumina and iron**, separation, 63
- precipitation by phenylhydrazine**, 44
- Aluminium and iron**, separation, 44, 95
- chloride**, compounds with organic substances containing oxygen, 294
- lead alloys**, 261
- tin alloys**, property of, 286
- welding**, 117
- zinc alloys**, 274
- "American Electro-chemical Society, Transactions"** (review), 16, 129
- American Electro-chemical Society**, 156
- Amidon**, retrogradation and regulation, 71
- retrograde**, formation and saccharification of, 101
- starch**, retrogradation of, 59
- Amines, aromatic**, action of chloracetamide on, 298
- cyclic**, 261
- primary**, at boiling-point, action of  $\text{PCl}_5$  on, 298
- di-alcoholic aromatic**, union of dinaphthopyryl salts with, 168
- primary and tertiary**, iodides of mercur-ammonium of, 34
- synthesis of cyclohexylamine and two other**, 155
- Aminoketones, aromatic**, intramolecular re-arrangement in derivatives of, 117
- Aminonitriles**, 59
- Ammeter, hot-wire**, for measuring very small alternating currents, 178
- Ammonia, alcoholic**, action of calcium on, 263  
and alkaline hydroxides, relative strengths of as measured by their action on cotarnine, 17  
and nitrate of silver compounds, 275
- liquid**, osmotic in, 59
- Ammonium aldehyde**, action of hydrocyanic acid on, 22
- metals**, action of carbonic anhydride on, 179
- thiocyanate**, 141
- "Analysis and Assaying, Metallurgical"** (review), 166
- Analytical operations**, use of organo-magnesian compounds in, 139
- Anhydride, carbonic**, action on ammonium metals, 179
- Aniline, aqueous solutions of**, action of carbon dioxide on in presence of nitrites, 59
- attempt to make take the form of amino-magnesian phosphate**, 35
- homologues**, hydrogenation, 310
- hydrochloride**, hydrolysis, 77
- hydrogenation**, 155
- Anilines, derivatives of highly substituted**, 81
- "Annuaire pour l'An 1904"** (review), 22
- Anode**, influence of the physical nature of on constitution of electrolytic peroxide of lead, 278
- Anodes, iridio-platinum**, used in electrolysis of alkaline chlorides, resistance of, 138
- Anthraccene oxidation**, electrolytic, 236
- Antraquinone**, action of phenylmagnesium bromide on, 130
- Antimony**, 173  
and antimony trisulphide, mixtures, 119  
and tellurium, separation, 168  
estimation, 299

- Antimony sulphide and bismuth protosulphide, fusibility of mixtures of, 12  
solutions in, cryoscopic examination, 311  
trisulphide and antimony, mixtures, 119  
Apparatus for measuring contents of human blood, 245  
for metrical study of stationary electric waves on spiral wires, 245  
for regulating the action of vacuum pumps, 191  
volumetric, accurate, 240  
Archibald, E. H., revision of the atomic weight of rubidium, 223  
and D. McIntosh, basic properties of oxygen, 295  
and J. W. Walker, ionisation and chemical combination in the liquefied halogen hydrides and hydrogen sulphide, 294  
Argon in gases of hot springs of Guadeloupe, 250  
Aromatic compounds obtained from the hydro-aromatic series, 93  
Arseniate, methyl-amino-magnesian, 35  
trimethylamino-magnesian, 35  
Arsenites, amino-magnesian, 35  
Arsenic and copper alloys, 200  
estimation, electrolytic, 272  
in all the organs of the animal economy, 298  
in bird's eggs, 262  
in organic matters, detection, 226  
in organism, Berthelot's calorimetric bomb to prove, 291  
medicinal, elimination and distribution of in the organism in the form of methyl arsenate of soda, 25  
sesquioxide, constitution, 30  
sulphide, colloidal solutions of, precipitation, 215  
systematic alcoylation of, 12  
yellow, 71  
Ascoli, M., electric osmosis in liquid ammonia, 59  
Ash in the human body, 72  
report on, 41, 54  
"Assaying and Analysis, Metallurgical" (review), 166  
Atomic weight, relation of specific heat to, 165  
weights, international, 305  
of oxygen and hydrogen, 261  
report of International Committee, 68, 164, 173  
Atoms, valence of, and negative electricity, connection between, 25  
Attwell, H. M., and M. O. Forster, borylcarbimide, 235  
Atwater, W. O., and F. G. Benedict, "Experiments on the Metabolism of Matter and Energy in the Human Body" (review), 261  
Auger, V., systematic alcoylation of arsenic, 12  
and M. Billy, alkaline earth manganate-manganates, 155  
Australian patent law, 167  
Austrophones, 245  
Ayrton, Mrs. H., origin and growth of ripple marks, 308  
Azotic compounds, 22  
with ortho-substituted alcohol functions, transformation into indazolic derivatives, 311
- BACTERIA, radio-activity of, 245  
Bacteriology, King's College, London, 12  
Bacterium, oxidising, 27  
Bagley, G., and T. H. Easterfield, resin acids of conifers, 259  
Balance, magnetic, 308  
Balances, chemical, new design for, 11  
Balch, D. M., catalogues gratis, 167  
"Baltimore Lectures" (review), 213  
Bamber, M. K., estimation of the adulterant in citronella oil, 21  
Barger, G., microscopic method of determining molecular weights, 69  
Barium, amide and nitride of, 34  
ammonium, action of gases on, 13  
and magnesium, sodium, calcium, and strontium peroxides, iodometry of, 96  
and radium, separation, 97  
law of thermic constants, and heat of formation of compounds of, 123  
poly-meta-lanthanate, 198  
Barkla, C. G., energy of secondary Röntgen radiation, 178  
Barral, E., chloruration of phenyl carbonate in presence of antimony chloride, 251  
in presence of iodine, 238  
Baskerville, C., elements, verified and unverified, 109, 121, 135, 150, 162, 170, 186, 194, 210  
and G. F. Catlett, contributions to the chemistry of the rare earths, 197  
and H. Holland, contributions to the chemistry of the rare earths, 184  
and G. F. Kunz, action of radium rays and ultra-violet light on minerals, 1  
and E. G. Moss, contributions to the chemistry of the rare earths, 172  
and R. Stevenson contributions to the chemistry of the rare earths, 158  
and J. W. Turrentine, contributions to the chemistry of the rare earths, 147  
Bauman mercury-vapour lamp, 156  
Baubigny, H., and G. Chavanne, new method of estimation of halogen elements in organic bodies, 82  
and P. Rivals, separation of iodine from alkaline halogen salts of iodine, chlorine, and bromine, 12  
Baudouin, A., electric osmosis in methyl alcohol, 237, 286  
Bauge, G., crystalline chromous tartrate, 297  
Bayley, T. (obituary), 310  
Beck, Mr., microscopes, 94  
Bequerel, H., "Recherches sur une Propriété Nouvelle de la Matière" (review), 155  
Béhal, A., isomer of borneol, 119  
and M. Sommelet, method of synthesis of aldehydes, 83  
Beilby, G. T., hard and soft states in metals, 307  
Beis, C., action of mixed organo-magnesium compounds on phthalimide and phenyl-phthalimide, 244  
Bellars, A. E., and R. S. Morrell, separation of  $\beta$ -crotonic acid from  $\alpha$ -crotonic acid, 140  
Benedict, F. G., and W. O. Atwater, "Experiments on the Metabolism of Matter and Energy in the Human Body" (review), 261  
Bennett, W., notes on non-homocentric pencils and the shadows produced by them, 57, 153  
Benzene in illuminating gas, determination, 14, 29  
in poisoning by coal-gas, 74  
Benzophenone chloride, action of sodium methoxide on, 272  
Benzoylaminomethylbenzophenones, transformation of dibenzoyltoluidines into isomeric, 176  
Benzylidene aniline, additive products of with ethyl acetoacetate and ethyl methylacetoacetate, 259  
chloride, action of sodium methoxide on, 272  
Berg, A., influence of hydriodic acid on oxidation of sulphurous acid, 258  
Berthelot, C., fermentation of the indigo plant, 296  
Berthelot, M., use of alternating currents in chemistry, 286  
Berthelot's calorimetric bomb to prove existence of arsenic in organism, 291  
Bertrand, G., existence of arsenic in bird's eggs, 262  
oxidation of gayacol by laccase, 59  
pressure regulator for fractional distillations, 239  
separator for fractional distillation, 239  
use of Berthelot's calorimetric bomb to prove existence of arsenic in organism, 291  
Beryllium and thorium, separation, 77  
and titanium, separation, 76  
and zirconium, separation, 77  
Beulaygue, L., sodium monosulphide as an indicator in the estimation of glucose by Fehling solution, 71  
Bevan, E. J., and C. F. Cross, hydrocellulose, 235  
Beveridge, P. J., liquid state, 169, 181  
theory of electrolytic dissociation, 82  
Billy, M., and V. Auger, alkaline earth manganate-manganates, 155  
Bird's eggs, arsenic in, 262  
Bismuth, 174  
and tannin compounds, 238  
estimation, 10  
in the form of chromate, iodometric estimation, 143  
arseniate, 87  
use as a means of separation in rare earth series, 52  
oxide, hydrated, action on acid isomers of gallic acid, 227  
phosphate, 87  
protosulphide and antimony sulphide, fusibility of mixtures of, 12  
protosulphide and silver sulphide, fusibility of mixtures, 12  
salts, crystallised, production, 87  
sub-nitrate, 87  
Blaise, E. E., alcoyl-allyl-ketones, 119  
allyl and propenyl-alcoyl ketones, 179, 275  
method of preparation of aldehydes, and methodic degradation of acids, 191  
Blanc, G., synthesis of  $\alpha\alpha$ -dimethylglutaric and  $\alpha\alpha$ -dimethyladipic acids, 168  
and M. Desfontaines, certain derivatives of  $\alpha$ -campholonic acid and  $\alpha$ -campholenic racemic acid, 191  
and A. Haller, new syntheses effected by means of molecules containing the methylene group associated with one or two negative radicals, 59  
"Bleaching, Electrical, and Hypochlorites" (review), 46  
Block, E., "Alfred Warner's Theorie des Kohlenstoffatoms und die Stereochemie der Kohlenstoffverbindungen" (review), 46  
Blondlot rays, application to chemistry, 234  
emitted during chemical reactions, origin, 274  
Blood, human, apparatus for measuring contents of, 245  
influence of chemical form under which an element is presented to the organism on rapidity of its passage into, 24  
Boardman, T. H., and W. French, "Practical Chemistry" (review), 214  
Bodlander, G., and O. Störbeck, contribution to the study of cuprous compounds, 59  
Bodroux, F., general method of synthesis of aldehydes, 191  
synthesis of aromatic aldehydes, 83  
Body, human, ash in, 72  
Bodtker, E., formation of chloroanilines, 287  
Boiling-points of homologous compounds, 265  
Bolduan, C., and A. Wassermann, "Immune Sera" (review), 310  
Bomb, calorimetric, to prove existence of arsenic in organism, 291  
Bone, W. A., and J. Drugman, action of ozone on ethane, 273  
and W. E. Stockings, slow combustion of ethane, 246  
J. J. Sudborough, and C. H. G. Sprankling, acid esters of methylsuccinic acid, 177  
Bonniksen, B., new dilatometer, 127  
Boone, W. T., "Safe" Course in Experimental Chemistry" (review), 261  
Boote well water, composition, 9  
Borneol, isomer of, 119  
Boraycarbimide, 235  
Bottone, S., "Radium and All About It" (review), 250  
Boucher, C., new method for attack of galenas and chalcopyrites, 56  
Bouchonnet, A., and C. Chabrie, preparation of iridium sesquiselelide, 34  
Boudouard, O., allotropic transformations of nickel steels, 130  
new method of determining critical-points of iron and steels, 54  
Boulough, R., formation of phosphorus sulphides in the cold, 130  
Bourion, F., and C. Matignon, general method of preparation of anhydrous chlorides, 179  
transformation of oxides and oxygen salts into chlorides, 203  
Bousfield, E. G. P., experiments with a new primary cell, 201  
note on determining accurately the percentage of ozone in gases not dissociated by moderate heat, 201  
W. R., purification of water by continuous fractional distillation, 141  
Bouveault, L., application of Grignard's reaction to halogen ethers of tertiary alcohols, 275  
Jecker Prize, 58  
purification and identification of alcohols, 251  
and G. Blanc, preparation of primary alcohols by means of corresponding amides, 94  
and A. Wahl, action of peroxide of nitrogen on isonitroso-malonie ethers, 311  
granular synthesis of the fatty aldehydes, 226  
nitro-isobutylene, 23  
preparation of diketonic ethers, 298  
reduction of  $\alpha$ -nitrostyrene, 23  
Brathwaite, J. O., "Year-book of Pharmacy" (review), 21  
Braun, G. and A., "DiGlossaire de Chimie Photographique" (review), 274  
Bread, examination of ancient samples, 199  
Brenans, F., new tri-iodine phenol, 34  
Briggs, S. H. C., ammoniacal double chromates and molybdates, 235

- Briggs, S. H. C., hexahydrated double chromates, 235
- Brissac, M., some amino-magnesian arseniates, 35
- and C. Porcher, attempts to make aniline and urea take the form of amino-magnesian phosphate, 35
- some amino-magnesian phosphates, 34
- "British Guiana, General Information with regard to the Gold, Diamond, and Forest Industries of" (review), 130
- "British Guiana, Report on Agricultural Work in Botanic Gardens and Government Laboratory at" (review), 274
- "British Patents for Inventions, Grant and Validity of" (review), 142
- Brochet, A., action of copper on chloric acid, 269
- basic copper chloride, 293
- electrolysis of alkaline and alkaline-earth chlorates with a copper anode, 293
- of chloric acid and chlorates, 206
- electrolytic solution of platinum, 274
- formation of basic salts of copper under influence of electrolysis, 279
- and J. Petit, influence of complex ions in electrolysis by alternating currents, 143
- use of alternating currents for electrolysis, 130
- Bromates, estimation, 95
- Bromine, action on 3:5-dichloro-1:1-dimethyl-delta 2:4-dihydrobenzene, 93
- alkaline halogen salts of, separation of iodine from, 12
- Bromophenol, action of magnesium and organo-magnesium compounds on, 262
- Brown, J. C., potable waters in South-West Lancashire, 6
- Brownson, H. W., volumetric method for the estimation of mercury fulminate, 303
- Brunk, A., estimation of ozone, 243
- Brunel, L., preparation of hydro-aromatic alcohols, 59
- Brushite, artificial production, 203
- Buffalo cow, sugar in milk of, 262
- Bullier, L. M., obtaining calcium carbide, 238
- Bunel, L. J., and C. Marie, estimation of persulphates, 113
- Burgess, C. H., and D. L. Chapman, nature of a solution of iodine in aqueous potassium iodide, 176
- photochemically active chlorine, 152
- H. K., and T. H. Page, composition of distilled oil of limes and a new sesquiterpene, 176
- Burke, Miss K. B., and F. G. Donnan, chemical dynamics of the alkyl iodides, 140
- Burosi, G., organo-mercuric compounds of salicylic acid, 120
- Butter-fat analysis, interdependence of the physical and chemical criteria in, 80
- CADMIUM** arsenide, 167
- salts, 71
- soluble, estimation, 114
- silver series of alloys, properties of, 86
- Cain, J. C., halogen derivatives of diphenyl and dihydroxydiphenyl, 18
- Calcium, action on alcoholic ammonia, 263
- strontium, barium, magnesium, and sodium peroxides, iodometry of, 96
- acetate, ignition of material with, 55
- Calcium carbide, obtaining, 238
- preparation, 191
- chloride, electrolysis, 297
- oxalate, precipitation of magnesium oxalate with, 146
- "Calculating Tables" (review), 214
- "Calendar, Imperial University of Tokyo" (review), 249
- Californian laurel, constituents of essential oil of, 224
- Callendar, H. L., projection of indicator diagrams of a petrol motor, 306
- Cambridge Scientific Instrument Company, vibrograph, 245
- Camphorones, cis- $\pi$ , of *d*- and *l*-hydriindamines, 19
- Campholenic derivatives, 119
- Camphor, ethylidene, 168
- monopoly in Japan, 250
- Capacities, measurement of small inductances and, 177
- Caprolyl thiocarbimide, 273
- Carbides, oxaloyl-ethylenic, 119
- Carbimides, optically active, 259
- "Carbon Atom, Alfred Werner's Theory of the, and the Stereochemistry of the Carbo-cyclic Compounds" (review), 46
- Carbon, action on quicklime at the temperature of fusing platinum, 119
- atomic weight, 297
- dioxide, action on aqueous solutions of aniline in presence of nitrites, 59
- on solutions of sodium nitrite, 150, 155
- disulphide solution, action of heat and light on mixtures of phosphorus sesquisulphide and sulphur in, 130
- monosulphide, gaseous, 25
- monoxide and oxygen, combining volumes of, 223
- sulphide, constants of, 83
- Carnot, A., "Traité d'Analyse des Substances Minérales" (review), 155
- Carques and Fonsses - Diacon, M.M., toxicity of nitro-prussiate of soda, 226
- volumetric estimation of alkaline nitroprussiates and of soluble salts of cadmium, 114
- Carré, P., etherification of phosphoric acid by glycerin, 34
- phosphoric ethers of glycerin, 70
- of glycol, 139
- Cartaud, G., and F. Osmond, meteoric iron, 34
- Caspian Sea, analysis of the combustible gas given off in the, 78
- Catalogues gratis, 167
- Cattlet, G. F., and C. Baakerville, contributions to the chemistry of the rare earths, 197
- Cavalier, J., silver and lead salts of monoalkylphosphoric acids, 203
- Cell, primary, experiments with, 201
- "Ceramic Calculations, Text-book of" (review), 274
- Cerium, 174
- and phenylhydrazine, 64
- behaviour toward organic bases, 15, 27
- Ceric earths, fractionation, 277
- Chabré, C., and A. Bouchonnet, preparation of iridium sesquiselenide, 34
- Chalcopyrites, attack of, 56
- Changes, intramolecular and originally reversible, extending over prolonged periods, 115, 196, 207
- Chapman, D. L., and C. H. Burgess, nature of a solution of iodine in aqueous potassium iodide, 176
- photochemically active chlorine, 152
- Charabot, E., and A. Hebert, influence of its surroundings on vegetable acidity, 227
- Charitchkof, K. V., analysis of the combustible gas given off in the Caspian Sea, 78
- Chassevant, A., preparation and properties of pure colloidal silver, 218
- Chattaway, F. D., dibenzoyl-chlorimide, 93
- Intramolecular rearrangement in derivatives of aromatic aminoketones, 117
- isomeric change of diacylanilides into acylamino ketones, 117
- and W. H. Lewis, isomeric change of diacylanilides into acylaminoketones, 176
- and J. M. Wadmore, derivatives of highly substituted anilines, 81
- Chaulmoogra seeds, constituents, 294
- Chavanne, G., ethers of isopyromucic acid, 22
- and H. Baubigny, estimation of halogen elements in organic bodies, 82
- Chelsea, South-Western Polytechnic, 24, 227
- "Chemical and Metallurgical Society of South Africa, Proceedings" (review), 46
- Chemical balances, new design for, 11
- combination and ionisation, 294
- Metallurgical, and Mining Society of South Africa, 71
- Society, 17, 67, 80, 92, 114, 140, 152, 175, 188, 220, 235, 245, 259, 271, 293
- President's address, 190
- report of Council, 188
- Chemicals, adulterated, 180
- "Chemistry, Aids to" (review), 201
- "Chemistry, Analytical, Short Manual of" (review), 47
- "Chemistry, Descriptive" (review), 237
- "Chemistry, Elementary Practical" (review), 21
- "Chemistry, Experimental, Safe Course in" (review), 261
- "Chemistry, General, Laboratory Outline of" (review), 154
- "Chemistry, Inorganic, Elements of" (review), 166
- "Chemistry, Inorganic, Karl Heumann's Directions for Lecture Experiments in" (review), 166
- "Chemistry, Inorganic, Principles of" (review), 261
- "Chemistry, Mineral, Industries of" (review), 46
- "Chemistry, Photographic, Dictionary of" (review), 274
- "Chemistry, Physical, Introduction to" (review), 214
- "Chemistry, Physical, Introduction to the Study of" (review), 154
- "Chemistry, Physical, Journal of" (review), 167
- "Chemistry, Practical" (review), 214
- Chemistry, application of Blondlot rays to, 234
- of the rare earths, 147, 158, 172, 184, 197
- position in medical curriculum, of University of London, 65
- use of alternating currents in, 286
- what physical chemistry has done for, 90, 105
- Chesteau, G., apparent diminution of energy of a dilute acid in presence of a neutral salt of this acid, 250
- Chloracetimide, action on aromatic amines, 298
- Chlorates, electrolysis of, 106
- estimation, 95
- Chlorides, anhydrous, preparation, 179
- detection in presence of bromides, 229
- Chlorides, transformation of oxides and oxygen salts into, 203
- Chlorine, action of light on, 152
- of silent discharge on, 296
- alkaline halogen salts of, separation of iodine from, 12
- and hydrogen, union, 152, 296
- density of, 58
- determination, 55
- in plants, 55
- methods for, 56
- photochemically active, 152
- Chloroanilines, formation, 287
- Chree, C., law of action between magnets, 284
- whirling and transverse vibrations of shafts, 153
- Chrétien and Guinchant, M.M., cryoscopic examination of solutions in antimony sulphide, 311
- Chromates and molybdates, ammoniacal double, 235
- hexahydrated double, 235
- Chromium and iron, separation by fused potassium nitrate, 183
- and vanadium, separation, 182
- estimation, colorimetric, 268
- Chrysanthemums, culture, chemical study of, 60
- Ciliocriche, 308
- Clarke, F. W., report of committee on atomic weights, 164, 173
- Clowes, F., and J. B. Coleman, "Elementary Practical Chemistry" (review), 21
- "Classification of Elementary Substances" (review), 225
- Cobalt and iron, separation, 280
- Coffgier, C., solubility of hard copals, 24
- Cohen, J. B., and P. Hartley, nitration products of isomeric dichlorobenzenes, 297
- and J. Marshall, reduction of 2:6-dinitrotoluene with hydrogen sulphide, 177
- and J. Miller, influence of substitution in nucleus on rate of oxidation of side chain, 80
- H. S. Raper, and J. T. Thompson, action of sodium hypochlorite on aromatic sulphonamides, 153
- Coherence and recoherence, 285
- Coleman, J. B., and F. Clowes, "Elementary Practical Chemistry" (review), 21
- College system and technical research, 230
- Collie, J. N., and Sir W. Ramsay, spectrum of radium emanation, 301
- Colloidal solutions, 202
- Colloids, precipitation of, 202
- Colouring matter of indigo, constitution, 238
- matters, some natural, 18
- triphenylmethane, 247
- Colours, calculation for colour sensatimeters, 212
- Colson, A., acetates of alkaline earths, 34
- application of Blondlot rays to chemistry, 234
- origin of the Blondlot rays emitted during chemical reactions, 274
- Combustion, calculating heats of, 22
- heat of nitrated organic compounds, 237
- Compounds, connection between volatility of, and chemical forces at play within the molecule, 241
- homologous, boiling-points, 265
- inorganic, systematic classification, 120
- nitrated organic, heat of combustion, 237
- optically active, influence of solvents on rotation, 297
- Conduche, A., and L. J. Simon, new reaction of aldehydes, 288
- Conifers, resin acids of, 250
- Constants, physical, at low pressures, 205, 217

- Copale, hard, solubility, 24  
 "Copper, Electrolytic Refining of" (review), 225  
 Copper, action on chloric acid, 269  
 and arsenic alloys, 200  
 anode, electrolysis of alkaline and alkaline-earth chlorates with, 293  
 estimation, iodometric, 288  
 influence on silvering of glass, 23  
 chloride, basic, 293  
 salts, basic, formation, 279  
 Coysus, G., and H. Wuyts, action of sulphur on organo-magnesian compounds, 227  
 Cowper-Coles, S. O., welding of aluminium, 117  
 Crepieux, P., and F. Reverdin, nitration of acetylalcol, 287  
 Crocker, J. C., and F. H. Lowe, picryl derivatives of urethane and thiourethane, 235  
 Crookes, Sir W., and J. Dewar, London water supply, 52, 88, 146, 211, 242, 304  
 Cross, C. F., and E. J. Bevan, hydrocellulose, 235  
 Crossley, A. W., aromatic compounds from hydroaromatic series, 93  
 Cunyngame, H. H., muffle and melting furnaces, 245  
 Cupel, magnesia, 304  
 Cuprous compounds, 59  
 Curie and Dewar, Profs., examination of sample of gas occluded in radium bromide, 85  
 Current, alternating, propagation along a telephone cable, 306  
 Currents, alternating, hot-wire ammeter for measuring very small, 178  
 instruments for measurement of large and small, 248  
 use for electrolysis, 130  
 in chemistry, 286  
 "Cyanide Process of Gold Extraction" (review), 142  
 "Cyaniding Gold and Silver Ores" (review), 249  
 Cyanogen estimation, 229  
 Cyclohexane and its chlorised derivatives, 298  
 Cyclohexanol and cyclohexanone, preparation from phenol, 33  
 Cyclohexylamine and two other amines, synthesis, 155
- DAHMER, G., and F. W. Kuster,** precipitation of colloidal solutions of sulphide of arsenic, 215  
 Daniel, K., and H. Leberle, estimation of iron in presence of zirconium, 311  
 Darling, C. N., thin-film electrolysis, 165  
 Davis, O. C. M., and F. E. Francis, action of nitrogen sulphide on organic substances, 93  
 Dawson, H. M., formation of peroxides in organic solvents, 152  
 and Ethel Elizabeth Goodson, formation of peroxides in nitrobenzene solution, 273  
 D'Anselmio, A., ratio between solubility of lime in presence of alkaline and caustification of alkaline carbonates, 298  
 solubility of hydrated sulphate of lime in solutions of sea-salt, 9  
 De Forcrand, M., zinc peroxides, 95  
 De Koninck, L., production of pure iodine, 311  
 De la Roche, B., phenolic urethanes of piperidine, 235  
 De Schulten, A., production of crystallised salts of bismuth, 87  
 research on dicalcic phosphate, 203  
 De Witteville, C., flame spectra of the alkaline metals, 150
- Dead Sea, principal cause of saltiness, 13  
 Deboureaux, L., estimation of chlorates, bromates, and iodates, 95  
 of nitrogen, 298  
 titration of manganese, 83  
 Defacoz, E., fluochlorides, fluobromides, and fluoiodides of the alkaline earth metals, 106  
 preparation of anhydrous and crystalline fluorides, 59  
 Delange, R., dichloromethenedioxypropylbenzene and propyl pyrocatechin carbonate, 143  
 and C. Moureu, amyl chlor-acrylic ethers, 226  
 decomposition of acetylenic acids by alkalis, 226  
 general observations in the acetylenic acids, 226  
 hydration of acetylenic acids, 226  
 new fatty acid,  $\gamma$ ,  $\gamma'$ ,  $\gamma''$ -trimethylbutyric acid, 226  
 Delépine, M., action of hydrocyanic acid on ammonium aldehyde and analogous combinations, 22  
 aminonitriles, 59  
 Demarçay, E., life and work of, 137  
 Deuliges, G., reactions of pinacolin and of pinacone, 35  
 Dennis, L. M., and J. G. O'Neill, determination of benzene in illuminating gas, 14, 29  
 Denso, P., resistance of iridio-platinum anodes used in electrolysis of alkaline chlorides, 138  
 Dervin, E., heat and light on mixtures of phosphorus sesquisulphide and sulphur in carbon disulphide solution, 130  
 Descudé, M., symmetric bichloromethyl oxide, 275  
 Desfontaines, M.,  $\alpha$ -substituted  $\beta$ -methyladipic acids, 107  
 and G. Blanc, derivatives of  $\alpha$ -campholytic acid and  $\alpha$ -campholenic racemic acid, 191  
 Dewar, J., physical constants at low temperatures, 205, 217  
 and Sir W. Crookes, London water supply, 52, 88, 146, 211, 242, 304  
 and Curie, Profs., examination of a sample of gas occluded in radium bromide, 85  
 and H. O. Jones, chemical reactions of nickel carbonyl, 68  
 Di-sodium tetra-lanthanate, 198  
 Diacylanilides, isomeric change into acylaminoketones, 117, 176  
 Diagrams, preparation of, 128  
 "Diamond, Gold, and Forest Industries of British Guiana" (review), 130  
 Diazobenzene and phenol, limit of union, 311  
 chloride, action on diphenylamine, 275  
 Dibenzoylchlorimide, 93  
 Dibenzoylchlorulidines, transformation into isomeric benzoylaminomethyl-benzophenones, 176  
 Dichlorobenzenes, nitration products of the isomeric, 297  
 Dichloromethenedioxypropylbenzene, 143  
 "Dictionary of Hygiene" (review), 22  
 "Dictionary of Photographic Chemistry" (review), 274  
 "Dictionary, Technological and Scientific" (review), 275, 303  
 Didymium orthophosphate, 299  
 salts containing phosphoric acid, absorption spectra of solutions of, 299  
 Diffraction and interference, 57  
 Dihydroxydiphenyl, halogen derivatives of, 18  
 Diketones,  $\beta$ -, 140
- Dilatometer, 127  
 Dinaphthopyranic series, 262  
 Dinitrotoluene, 2 : 6, reduction with hydrogen sulphide, 177  
 Dipentene, synthesis, 233  
 Diphenyl, halogen derivatives of, 18  
 Diphenylamine, action of diazobenzene chloride on, 275  
 Diphenylanthracene, 310  
 anhydride, symmetric, 310  
 "Directory of Paper Makers of the United Kingdom" (review), 193  
 "Directory, Paper Trade" (review), 168  
 "Dispensing Made Easy" (review), 142  
 Dissociation, electrolytic, theory of, 31, 37, 82, 202  
 "Distillation, Fractional" (review), 129  
 Distillation, fractional, separator for, 239  
 pressure regulator for, 239  
 Distilleries, tar, accidents in, 35  
 Diurides, 130  
 Divers, E., constitution of nitric peroxide, 18  
 peroxylaminesulphonic acid, 18  
 Dixon, A. E., caproylthiocarbimide, 273  
 organic phosphorus compounds, 116  
 W. E., and O. Inchley, cilio-scribe, 308  
 Dobbie, J. J., A. Lauder, and C. K. Tinkler, relative strengths of alkaline hydroxides and of ammonia as measured by their action on cotarnine, 17  
 Doby, G., action of calcium on alcoholic ammonia, 263  
 Documents, filing, Stolsenberg system, 269  
 Donnan, F. G., and Miss K. B. Burke, chemical dynamics of alkyl iodides, 140  
 Doran, R. E., tautomeric character of the acyl thiocyanates, 92  
 Dreaper, W. P., technical research and the college system, 230  
 Drugman, J., and W. E. Stockings, action of hydrogen sulphide on formaldehyde and acetaldehyde solutions, 260  
 and W. A. Bone, action of ozone on ethane, 273  
 Drugs, adulterated, 180  
 "Drugs, Their Production, Preparation and Properties" (review), 142  
 Drying machine, vacuum, 312  
 Du Jassoneix, Binet, and H. Moisan, M.M., density of chlorine, 58  
 Dubreuil, L., action of bromosuccinic and bibromosuccinic acid on pyridic and quinolic bases, 34  
 Duddell, W., some instruments for the measurement of large and small alternating currents, 248  
 Dufour, A., apparent volatilisation of silicon in hydrogen, 286  
 formation of hydrogen silicide, 265  
 reduction of silicon by hydrogen, 274  
 Dunstan, A. E., viscosity of liquid mixtures, 260  
 Duval, H., nitric ethers of acid alcohols, 35, 59, 227  
 Dyer, E., and F. W. B. Sbrivell, "Manuring of Market Garden Crops" (review), 95
- EAKLE, A. S.,** "Mineral Tables for the Determination of Minerals by their Physical Properties" (review), 190  
 Earle, rare, series, use of bismuth as a means of separation in, 52
- Earths, alkaline, acetates of, 34  
 ceric, fractionation, 277  
 rare, chemistry of, 147, 158, 172, 184, 197  
 Easterfield, T. H., and G. Bagley, resin acids of the conifers, 259  
 and O. Silberrad, studies on ethyl carboxyglutarate, 259, 296  
 Ehler, E., and E. Kiroevenagel, use of hydroxylamine and hydrazine in qualitative analysis, 203  
 Eder, J. M., and E. Valenta, invariability of the wave-lengths in the spark and arc spectrum of zinc, 98  
 Edwards, V., scale for chemical valuation of soils, 197  
 rapid soil analysis, 183  
 Egge, bird's, arsenic in, 262  
 Ehrhfeld, R., speed of the reaction between permanganate of potassium and oxalic acid, 96  
 Electric accumulator, 287  
 accumulators, manufacture of, regulations for, 35  
 furnace, 307  
 sparks, distribution and spectra of metallic vapours in, 235  
 waves, stationary, on spiral wires, apparatus for metrical study of, 245  
 "Electricity, &c., Subject-list of Works on, in the Library of the Patent Office" (review), 310  
 Electricity, negative, and the valence of atoms, connection between, 25  
 "Electro-technics, &c., Subject-list of Works on, in the Library of the Patent Office" (review), 310  
 Electrolysis, applications to separation of metals from one another, 110, 118, 126  
 by alternating currents, influence of complex ions in, 143  
 of alkaline chlorides, resistance of iridio-platinum anodes used in, 138  
 solutions, hydrates in concentrated aqueous, 157  
 thin-film, 165  
 use of alternating currents for, 130  
 "Electrolytic Preparations" (review), 201  
 Electroscopes, portable, 128  
 Element, influence of chemical form under which it is presented to organism on rapidity of its passage into the blood, 24  
 Elements and compounds, 220  
 halogen, in organic bodies, estimation, 82  
 verified and unverified, 109, 121, 135, 150, 162, 170, 186, 194, 210  
 why are some so much more abundant than others, 47, 58, 118  
 "Elementary Substances, Classification" (review), 225  
 Elba, K., "Electrolytic Preparations" (review), 201  
 Elliott and Sons, vacuum machinery, 288  
 Emanation substance, 267  
 Eranium, 267  
 "Energy and Matter in the Human Body, Experiments on Metabolism of" (review), 261  
 Engelhardt, V., "Hypochlorite und Elektrische Bleiche" (review), 46  
 and J. W. Richards, "Electrolysis of Water" (review), 214  
 and T. Ulke, "Die Elektrolytische Raffination des Kupfers" (review), 225  
 Knock, F., colour photographs of living insects, 309  
 "Entropy" (review), 225



- Epichlorhydrine, action on sodium acetylacetone, 59  
 Epinephrine, constitution, 92  
 Erdmann, H., constitution of sesquioxide of arsenic, 30  
 and V. Unruh, yellow arsenic, 71  
 Esters, acid, of methylsuccinic acid, 177  
 of  $\beta$ -ketonic and  $\beta$ -aldehydic acids, optically active, 21  
 oxamic, imino-ethers and allied compounds corresponding with, 293  
 Etard, A., life and work of Eugene Demarçay, 137  
 Ethane, action of ozone on, 273  
 combustion, slow, 246  
 Ethanol and vanadic acid, colour reactions of, 82  
 Ether, chloroacetylacetic, 143  
 homoallantoic 130  
 isopropyl, conversion of isopropyl alcohol into, 260  
 methylic, of acetol, 231  
 oxides, halogen, 251  
 preparation by means of magnesium compounds and halogen methylic ethers, 215  
 Ethers, acetylenic, condensation with alcohols, 107  
 amylochloracrylic, 226  
 and olefin, biochemical synthesis, 130  
 diketonic, preparation, 298  
 halogen, of tertiary alcohols, application of Grignard's reaction to, 275  
 isonitrosomalonic, action of peroxide of nitrogen on, 311  
 nitric, of acid alcohols, 33, 59, 227  
 of isopropylmucic acid, 22  
 phosphoric, of glycerin, 70  
 of glycol, 130  
 Ethyl acetate, additive products of benzylidene aniline with, 259  
 bromocaroxyglutarate, action on ethyl sodiocaroxyglutarate 296  
 carboxylglutamate, formation, 296  
 carboxylglutarate, 259, 296  
 $\beta$ -iodopropionate, action on ethyl diiodioethanetetra-carboxylate, 176  
 methylacetate, additive products of benzylidene aniline with, 259  
 sodiocaroxyglutarate, action of ethyl bromocaroxyglutarate on, 296  
 of halogens on, 259  
 tartrate, rotation values at different temperatures, 259  
 Ethylidene camphor, 168  
 Europium, 179  
 Everett, J. D., normal piling as connected with Osborne Reynolds's theory of universe, 212  
 "Extra Pharmacopœia" (review), 309
- FARADAY** lecture, 220  
 Society, 117, 165, 200, 236, 307  
 Fat, butter, analysis, interdependence of physical and chemical criteria in, 80  
 Fats, thermostat for use in connection with the refractometric examination of, 80  
 Fatty series, halogen derivatives of, action of reduced nickel on in presence of hydrogen, 143  
 Fawcitt, C. E., decomposition of alkylureas, 273  
 relation between chemical composition of organic substances and density of their solutions, 117  
 studies in viscosity, 236  
 Fermentation of the indigo plant, 296  
 Fernbach, A. L. Maquenne, and J. Wolff, retrogradation and coagulation of amion, 71  
 Filing documents, Stolzenberg system, 269
- Findlay, A., freezing-point curves of dynamic isomerides, 141  
 "Phase Rule and its Applications" (review), 201  
 "Fire and Explosion Risks" (review), 286  
 Fleming, J. A., apparatus for metrical study of stationary electric waves on spiral wires, 245  
 hot-wire ammeter for measuring very small alternating currents, 178  
 measurement of small inductances and capacities, 177  
 model illustrating the propagation of an alternating current along a telephone cable, 306  
 Fluobromides, fluochlorides, and fluoiodides of alkaline-earth metals, 106  
 Fluorides, anhydrous and crystalline, preparation, 59  
 Fluorine, 173  
 and oxygen, causes of analogy between, 49  
 density, 202  
 Flury, F., and A. Gutbier, compounds of sulphur and tellurium, 131  
 Fontana, A., and F. M. Perkin, electrolytic oxidation of anthracene, 236  
 Fozzes-Diacon and Carquet, M.M., toxicity of nitroprussiate of soda, 226  
 volumetric estimation of the alkaline nitroprussiates, and of soluble salts of cadmium, 114  
 Ford, J. S., hydrolysis of starch by diastase, 247  
 "Forest, Diamond, and Gold Industries of British Guiana, General Information with regard to the" (review), 120  
 Formaldehyde and  $\alpha$ -tetrahydro- $\beta$ -naphthylamine, reaction between, 217  
 atmospheric, estimation, 311  
 polymers of, 298  
 solution, action of hydrogen sulphide on, 260  
 Forster, M.O., isonitrosocamphor, 295  
 and H. M. Attwell, bornylcarbidide, 235  
 and Frances Mary Gore Mickelthwait, action of nitrogen peroxide on  $\alpha$ -nitrocumaphene, 92  
 Fosse, R., new dinaphthopyranic phenols, 119  
 researches on the dinaphthopyranic series, 262  
 union of dinaphthopyryl salts with dialcoholic aromatic amines, 168  
 Fournelle, E., certain aminoalcohols of tertiary alcoholic function, 203  
 Fox, J. J., and J. T. Hewitt, studies in the acridine series, 70  
 Francis, F. E., and O. C. M. Davis, action of nitrogen sulphide on organic substances, 93  
 and Miss Millicent Taylor, additive products of benzylidene aniline with ethylacetate, and ethyl methylacetate, 259  
 Francois, M., iodides of mercurammonium of primary and tertiary amines, 34  
 Frape, G. S., report on ash, 41, 54  
 Freezing-point curves of dynamic isomerides, 141  
 French, W., and T. H. Boardman, "Practical Chemistry" (review), 214  
 Freunder, F., reduction of nitrobenzole acetals and acids, 119  
 researches on azotic compounds, 22  
 transformation of azotic compounds with ortho-substituted alcohol function into indazylid derivatives, 311
- Friend, J. A. N., estimation of hydrogen peroxide in presence of potassium persulphate by means of potassium permanganate, 177  
 Friiswell, R. J., intramolecular and originally reversible changes extending over prolonged periods of time, 115, 196, 207  
 Furfuraldehyde, condensation with sodium succinate, 80  
 Furnace, electric, 307  
 Furnaces, muffle and melting, 245  
 Fusion with sodium hydroxide, 56
- GADD, H. W.**, "Drugs—their Production, Preparation, and Properties" (review), 124  
 Gadolinium oxide, preparation of pure, 232  
 Galactose, mutarotation, 246  
 Galenas, attack of, 56  
 Garrett, C. A. E., and P. E. Shaw, coherence and recoherence, 285  
 Gas, acetylene, application to heating of germination stoves, 215  
 coal, part played by benzene in poisoning by, 74  
 combustible, given off in the Caspian Sea, analysis, 78  
 illuminating, determination of benzene in, 14, 29  
 occluded in radium bromide, examination, 65  
 Gases, action on barium-ammonium, 13  
 chimney, estimation of unburnt products in, 61  
 not dissociated by moderate heat, determining percentage of ozone in, 201  
 permanent, determination of molecular weight of, 207  
 Gauthier, D., compounds of saccharose with certain metallic salts, 59, 179  
 Gautier, A., degree of accuracy attained in detection of traces of arsenic in organic matters, 226  
 normal existence of arsenic in all the organs of the animal economy, 298  
 Gayaccol, oxidation by laccose, 59  
 Genavresse, P., action of paraformaldehyde on the sesquiterpenes, 298  
 Gerard, J., "The Old Riddle and the Newest Answer" (review), 225  
 Getman, F. H., and H. C. Jones, existence of hydrates in concentrated aqueous solutions of electrolytes, 157  
 Gibello, M., and A. Seyewetz, new polymers of formaldehyde, 298  
 synthesis of sugars from trioxymethylene and sodium sulphite, 86  
 Gleele, F., emanation substance, 267  
 Giraud, M., behaviour of phenolphthalein in presence of bicarbonates and alkaline carbonates, 27  
 Glass silvering, influence of copper on, 23  
 Glazebrook, R. T., Address to Physical Society, 93  
 diffraction theory of the microscope, 213  
 Glucium chloride, heat of formation, 176  
 Glucose, estimation by Fehling solution, sodium monosulphide as an indicator in, 71  
 mutarotation, 246  
 Glycerides, higher, 175  
 Glycerin, phosphoric ethers of, 70  
 Glycol, phosphoric ethers of, 130  
 Glycols, primary  $\alpha$ -, transformation into corresponding aldehydes, 59
- Godefroy, L., and E. Varenne, ethylic alcohol hydrates, 23  
 hydrates of methylic alcohol and acetone, 244  
 "Gold and Silver Ores, Cyaniding" (review), 249  
 "Gold, Diamond, and Forest Industries of British Guiana, General Information with regard to the" (review), 130  
 "Gold Extraction, Cyanide Process of" (review), 142  
 Gold analysis, electrolytic, 165  
 colloidal, 262  
 aqueous solution of, 143  
 Goodrich, W. F., "Refuse Disposal and Power Production" (review), 285  
 Goodson, Ethel Elizabeth, and H. M. Dawson, formation of periodides in nitrobenzene solution, 273  
 Goodwin, W., and L. Maquenne phenylurethanes of sugar, 179  
 Gordon, J. W., high-power microscopy, 245  
 Gorgeu, A., hausmannites from Sweden, 211  
 Gornall, F. H., and F. B. Power, constituents of chaumoogra seeds, 294  
 constitution of chaumoo-gric acid, 293  
 gynocardin, 295  
 Goellings, N., and A. Warner, carbonatopentamine cobaltic salts, 275  
 Granger, A., cadmium arsenide, 167  
 Green, A. B., radio-activity of bacteria, 245  
 A. G., and A. G. Perkin, constitution of phenolphthalein, 141  
 Grignard, V., action of magnesium and organo-magnesium compounds on bromophenestol, 262  
 preparation of diethylacetate of methyl, 298  
 primary phenylethyl alcohol, 298  
 synthesis of tertiary alcohols by means of organo-magnesium compounds, 95  
 Grignard's reaction, application to halogen ethers of tertiary alcohols, 275  
 Grossmann, H., salts of cadmium, 71  
 Guillet, L., constitution and properties of silicon steels, 34  
 of vanadium steels, 130  
 Guinchant and Chretien, M.M., cryoscopic examination of solutions in antimony sulphide, 311  
 Gunz, M., La Case Prize, 58  
 and M. Mentral, action of gases on barium-ammonium, 13  
 amide and nitride of barium, 34  
 Gutbier, A., action of phenylhydrazine on oxidised compounds of selenium and tellurium, 131  
 aqueous solution of colloidal gold, 143  
 of colloidal selenium, 143  
 examination of colloidal sulphides, 59  
 gravimetric estimation of tellurium by means of hypophosphorous acid, 143  
 quantitative separation of tellurium and antimony, 168  
 and F. Flury, compounds of sulphur and tellurium, 131  
 and E. Rohn, estimation of selenium, 311  
 Guye, F. A., determination of molecular weight of permanent gases, 297  
 and E. Mallet, atomic weights of oxygen and hydrogen, 261  
 Guyot, A., and A. Haller, action of phenyl-magnesium bromide on anthraquinone, 129

- Guyot, A., and A. Haller, diphenyl-anthracene and symmetric diphenyl anthracene dihydride, 310  
and Stoehling, M.M., derivatives of tetramethyldiaminophenyl-oxanthranol, 107  
Gynocardin, 295
- HABER, F.**, and M. Stoecker, "Praktische Übungen zur Einführung in Die Chemie" (review), 154  
Haberg, E., electrolytic estimation of thallium, 311  
Hackford, J.E., and H. J. S. Sand, electrolytic estimation of minute quantities of arsenic, 272  
Hæmatin, separation of the principles decomposing peroxide of hydrogen contained in the, 298  
Haga, T., peroxyaminesulphonates, 17  
Halides of alkali metals, action of radium rays on, 246  
Haller, A., preparation of alcoyl and alcoylidonic derivatives of cyclic ketones, 286  
and G. Blanc, syntheses effected by means of molecules containing the methylene group associated with negative radicals, 59  
and A. Guyot, action of phenylmagnesium bromide on anthraquinone, 130  
diphenylanthracene and symmetric diphenylanthracene dihydride, 310  
Halogenes, action on ethylsodio-carboxylglutarate, 259  
Hamonet, J., halogen ether oxides, 251  
preparation of ether oxides by means of magnesium compounds and halogen methylic ethers, 215  
Hamy, M., spectrum of zinc, 257  
Hann, A. C. O., and A. Lapworth, additive compounds of unsaturated cyclic ketones with hydrogen cyanide, 152  
optically active esters of  $\beta$ -ketonic and  $\beta$ -aldehydic acids, 21  
Hannay, J. B., higher glycerides, 175  
Hanriot, M., colloidal gold, 262  
Hanson, E. K., and R. S. Morrell, resolution of  $\alpha$ -dihydroxybutyric acid into its optically active constituents, 93  
Hartley, F., and J. B. Cohen, nitration products of isomeric dichlorobenzenes, 297  
Hartwell, B. L., behaviour of cerium, lanthanum, neodymium, praseodymium, thorium, and zirconium toward organic bases, 15, 27  
Harvey, A. W., phenyldimethylallylammonium compounds, 177  
Hausermannites from Sweden, 211  
Heat, action on hydroxy-carboxylic acids, 81, 294  
on mixtures of phosphorus sesquisulphide and sulphur in carbon disulphide solution, 150  
Hebert, A., and E. Charabot, influence of its surroundings on vegetable acidity, 227  
and G. Truffaut, chemical study of culture of chrysanthemums, 60  
Helbronner, A., derivatives and products of condensation of  $\beta$ -oxy-naphthoic aldehyde, 287  
Helium, production from radium, 255, 266  
Henri, V., and A. Mayer, colloidal solutions, 202  
Henriet, H., estimation of atmospheric formaldehyde, 311  
formic aldehyde in atmospheric air, 120  
Henry, L., methylic ether of acetol, 251  
trichlor-isopropyl alcohol, 106  
T. A., "Aids to Chemistry" (review), 201  
Herz, W., simultaneous volumetric estimation of boric acid and strong acids, 60  
Hewitt, J. T., and J. J. Fox, studies in the acridine series, 70  
J. Kenner, and H. Silk, bromination of phenolic compounds, 272  
"Heumann's, Karl, Directions for Lecture Experiments in Inorganic Chemistry" (review), 166  
Hibbert, H., and J. J. Sudborough, estimation of hydroxyl radicals, 19  
W., magnetic balance, 308  
Hiora, A. J., alloys of copper and arsenic, 200  
Holland, H., and C. Barker, contributions to the chemistry of the rare earths, 184  
Holland, A., analysis of commercial nickel, 45  
applications of the theory of electrolysis to separation of metals from one another, 110, 118, 125  
influence of physical nature of anode on constitution of electrolytic peroxide of lead, 278  
Holmes, J., and T. E. Thorpe, estimation of methyl alcohol in presence of ethyl alcohol, 18  
Holroyd, G. W. F., magnesium oxybromide, 115  
Homfray, D., and C. T. Kingzett, "Dictionary of Hygiene" (review), 22  
House of Commons, ventilation, 168  
Howard, D., address to the Institute of Chemistry, 128  
Howgrave-Graham, R. P., "X-rays Simply Explained" (review), 190  
"Human Body, Experiments on the Metabolism of Matter and Energy in" (review), 261  
Hydrates in concentrated aqueous solutions of electrolytes, 157  
of methylic alcohol and acetone, 244  
Hydrazine, use in qualitative analysis, 203  
hydrate, vapour density of, 223  
Hydrides, halogen, and organic compounds, additive compounds of, 295  
ionisation and chemical combination in the liquefied, 294  
Hydrindamines, *d*-, and *l*-, cis-camphanates of, 19  
Hydroaromatic series, aromatic compounds obtained from, 93  
Hydrocarbides, aromatic, action of sulphur and selenium on organo-magnesium compounds of mono- and di-halogen, 251  
Hydrocellulose, 117, 235  
Hydrogen and chlorine, union, 152, 296  
and nickel, finely-divided, direct reduction of aromatic halogen derivatives of, 119  
atomic weight, 261, 297  
compressibility between one atmosphere and half an atmosphere of pressure, 86  
reduction of silicon by, 274  
solid, densities of, 205, 217  
formation at low temperature, 145  
volatilisation of silicon in, 286  
cyanide, additive compounds of unsaturated cyclic ketones with, 152  
peroxide, estimation in presence of potassium persulphate, 177  
separation of the principles decomposing, contained in the hæmatin, 298  
silicide, formation, 261, 286  
Hydrogen sulphide, action on formaldehyde and acetaldehyde solutions, 260  
ionisation and chemical combination in the liquefied, 294  
Hydroxylamine, action of, 22  
use in qualitative analysis, 203  
Hydroxylaminotrisulphonates, 17  
"Hygiene, Dictionary of" (review), 22  
"Hypochlorites and Electrical Bleaching" (review), 46  
Hypo-sulphite, sodic, and iodine, preparation of titrated solutions of, 311
- IGNITION** of material with calcium acetate, 55  
with sodium carbonate, 56  
Ikeda, K., and J. Sakurai, international atomic weights, 505  
"Immune Sera" (review), 310  
Imino-ethers and allied compounds corresponding with the substituted oxamic esters, 293  
Ince, J., "Latin Grammar of Pharmacy" (review), 46  
Inchley, O., and W. E. Dixon, cilioscirbe, 308  
Indazolic derivatives, formation, 22  
Indicator, new, 262  
Indigo, colouring-matter of, 238  
plant fermentation, 296  
Inducance, small, standard of, 177  
Inducances and capacities, measurement of small, 177  
Induction, action of temperature on period of, 152  
Insects, living, colour photographs of, 309  
Institute of Chemistry, 47, 78, 83, 128, 192, 239  
Interference and diffraction, 57  
Intramolecular changes, continuous, extending over prolonged periods, 115, 196, 207  
"Inventions, Grant and Validity of British Patents for" (review), 142  
Iodates, estimation, 95  
Iodides of mercur-ammonium of primary and tertiary amines, 34  
Iodine and sodic hypsulphite, preparation of titrated solutions of, 311  
pure, production, 311  
separation from alkaline halogen salts of iodine, chlorine, and bromine, 12  
solution in aqueous potassium iodide, 176  
Ionisation and chemical combination, 294  
Ions, complex, influence in electrolysis by alternating currents, 143  
Iridium sesquiselenide, preparation, 34  
Iron, 173  
and alumina, separation, 63  
and aluminium, separation, 44, 95  
and chromium, separation, 183  
and cobalt, separation, 280  
and nickel, separation, 280  
and titanium, separation, 63  
and zirconium, separation, 63  
estimation in presence of zirconium, 311  
formation of peroxides in the case of, 203  
meteoric, 34  
organisms in Lancashire waters, 8  
Irons, determining critical-points of, 34  
Isomerides, dynamic, freezing-point curves of, 141  
Isopilocarpine, fusion with caustic potash, 81
- JACKSON, H.**, phosphorescent materials, 245  
Jackson, H. A., thermo-chemistry of electrolytic dissociation, 202  
W., "Text-book of Ceramic Calculations" (review), 274  
Japan, camphor monopoly in, 259  
Joffrin, H., application of acetylene gas to heating of germination stoves, 215  
John J. Griffin and Sons, Ltd., accurate volumetric apparatus, 240  
Jones, C., detection of chlorides in presence of bromides, 219  
H. C., "Elements of Inorganic Chemistry" (review), 166  
what physical chemistry has done for chemistry, 90, 105  
and F. H. Getman, existence of hydrates in concentrated aqueous solutions of electrolytes, 157  
H. O., optically active asymmetric nitrogen compounds, 69  
and J. Dewar, chemical reactions of nickel carbonyl, 68  
Jordie, E., and E. H. Kanter, silicic acid, 251, 263  
Joseph, A. F., and J. E. Mackenzie, action of sodium methoxide and its homologues on benzophenone chloride and benzylidene chloride, 272  
Jowett, H. A. D., constitution of epinephrine, 92  
fusion of isopilocarpine with caustic potash, 81  
Just, A., complex double salt of manganous and tungstic acid, 285  
Julian, H. F., and E. Smart, "Cyaniding Gold and Silver Ores" (review), 249
- KANTER, E. H.**, silicic acid and the alkaline and alkaline-earth silicates, 23  
and E. Jordie, silicic acid, 251, 263  
Kelvin, Lord, "Baltimore Lectures" (review), 213  
Kenner, J. J., T. Hewitt, and H. Silk, bromination of phenolic compounds, 272  
Kerr, R., and A. Smith, micro-photography, 245  
Ketones, alcoyl-allyl-, 119  
allyl- and propenyl-alcoyl-, 179, 275  
boiling-points, 265  
cyclic, additive compounds of unsaturated with hydrogen cyanide, 152  
preparation of alcoyl and alcoylidonic derivatives of, 286  
Kingdon, J. C., why are some elements so much more abundant than others? 58  
Kingzett, C. T., and D. Homfray, "Dictionary of Hygiene" (review), 22  
Kipping, F. S., cis-camphanates of *d*- and *l*-hydrindamines, 19  
organic derivatives of silicon, 81  
and A. H. Salway, arrangement in space of groups combined with tervalent nitrogen atom, 116  
and F. Tutin, four optically isomeric *l*-menthylamines and their salts, 20  
Kiroevenagel, E., and E. Ebler, use of hydroxylamine and hydrazine in qualitative analysis, 203  
Kling, A., methyl acetate, 251  
and M. Viard, differentiation of primary, secondary, and tertiary alcohols of fatty series, 287  
Knight, N., precipitation of magnesium oxalate with calcium oxalate, 146  
Koppel, J., conditions of formation and solubility of cupro-sodic sulphate, 224

- Kouriloff, V., compounds of nitrate of silver with ammonia, 275
- Krauss, L., and E. Rupp, iodometric estimation of copper by means of xanthate of potassium, 283
- Krypton, 174
- Kühling, O., "Karl Heumann's Anleitung zum Experimentieren bei Vorlesungen über Anorganische Chemie" (review), 166
- Kunz, G. F., and C. Baskerville, action of radium rays and ultra-violet light on minerals, 1
- Kuster, F. W., and G. Dahmer, precipitation of colloidal solutions of sulphide of arsenic, 215
- L**ABY, T. H., separation of iron from nickel and cobalt by lead oxide, 280
- Lacombe, H., fractionation of the ceric earths, 277
- G. Urbain, europium, 179
- preparation of samaria and atomic weight of samarium, 277
- use of bismuth as a means of separation in rare earth series, 52
- Lactone, oxycrotonic, 262
- Lake, H. B., what may result from the study of radium, 47
- Lamp, mercury-vapour, Bastian, 156
- Lander, G. D., imino-ethers and allied compounds corresponding with substituted oxamic esters, 293
- Landolt, H., and J. H. Long, "Optical Rotating Power of Organic Substances" (review), 129
- Lanthanates, 197
- Lanthanum, 173
- behaviour toward organic bases, 15, 27
- Lanthanum-alums, 172
- Lapworth, A., and A. C. O. Hann, additive compounds of unsaturated cyclic ketones with hydrogen cyanide, 152
- optically active esters of  $\beta$ -ketonic and  $\beta$ -aldehydic acids, 21
- Larmor, J., absence of effects of motion through the ether in relation to constitution of matter, 284
- "Latin Grammar of Pharmacy" (review), 46
- Lauder, A. J., J. J. Dobbie, and C. K. Tinkler, relative strengths of alkaline hydroxides and of ammonia, 17
- Laurel, Californian, essential oil of, constituents, 224
- Lauth, C., triphenylmethane colouring matters, 297
- Le Sueur, H. R., action of heat on hydroxycarboxylic acids, 81, 294
- Lead-aluminium alloys, 261
- Lead bioxide, use in analysis, 26
- peroxide, electrolytic, influence of physical nature of anode on constitution of, 278
- salts of monoacetylphosphoric acids, 203
- "Leather Manufacture, Principles of" (review), 166
- Lebeau, P., dissociation of alkaline carbonates, 59
- Leberle, H., and K. Daniel, estimation of iron in presence of zirconium, 311
- Leclerc, A., method of separation of aluminium and iron by use of formic acid, 95
- Lees, F. H., derivatives of umbellulone, 224
- and F. B. Power, constituents of essential oil of Californian laurel, 224
- Leibmann, A., vacuum drying machinery, 312
- Lemout, P., action of  $\text{PCl}_5$  on cyclic primary amines at boiling-point, 298
- calculating heats of combustion, 22
- calculation of heat of combustion of nitrated organic compounds, 257
- experimental researches relative to certain cyclic amines, 261
- phospho-nitrated bases, 215
- Lenormand, C., estimation of organic matter in water, 219, 229
- Lespiau, M., chloroacetylacetic ether, 143
- oxycrotonic lactone and  $\gamma$ -substituted crotonic acids, 262
- Lewis, W. H., and F. D. Chittaway, isomeric change of diacylanilides into acylaminoketones, 176
- Light, action on chlorides, 152
- on mixtures of phosphorus sesquisulphide and sulphur in carbon disulphide solution, 130
- ultra-violet, action on minerals, 1
- Lime, ratio between solubility of and caustification of alkaline carbonates, 298
- sulphate, hydrated, solubility in solutions of sea salt, 9
- Limes, oil of, composition of a distilled, 176
- Lindet, L., examination of ancient samples of bread, 199
- inversion of sugar, 156
- Liquid state, 169, 181
- Locke, M., systematic classification of inorganic compounds, 120
- Locquin, R., method of identifying the fatty acids, 311
- London, King's College, 12
- University, position of chemistry in medical curriculum of, 65
- water supply, 52, 89, 146, 211, 242, 304
- Long, J. H., and H. Landolt, "Optical Rotating Power of Organic Substances" (review), 129
- Low, W. H., estimation of adulterant in citronella oil, 12
- Lowe, F. H., and J. C. Crocker, picryl derivatives of urethane and thiourethane, 235
- Lowry, T. M., mutarotation of glucose and of galactose, 246
- Lumière, A. L., and F. Perrin, action of chloracetamide on some aromatic amines, 298
- Lumsden, J. S., new design for chemical balances, 11
- M**CDOWALL, J., volumetric estimation of cyanogen, 229
- McIntosh, D., and E. H. Archibald, basic properties of oxygen, 295
- and J. W. Walker, ionisation and chemical combination in liquefied halogen hydrides and hydrogen sulphide, 294
- J. G., preparing Venetian red, 297
- McKenna, C. F., testing wood treated to resist fire, 32, 40
- McKenzie, A., esterification of  $\gamma$ -mandelic acid by menthol and borneol, 116
- Mackenzie, J. E., and A. F. Joseph, action of sodium methoxide and its homologues on benzophenone chloride and benzylidene chloride, 272
- Macleod, W. A., and C. Walker, "Metallurgical Analysis and Assaying" (review), 166
- Machinery, vacuum, 288
- Magnesia cupel, 304
- Magnesium, action on bromophenol, 262
- and nickel compounds, 235
- Magnesium oxalate, precipitation with calcium oxalate, 146
- oxybromide, 115
- peroxides, iodometry of, 96
- "Magnetism, &c., Subject-list of Works on in Library of the Patent Office" (review), 310
- Magnetograph, quartz-thread vertical force, 127
- Magnetostatic field, stresses in a, 128
- Magnets, law of action between, 284
- Mallie, A., and P. Sabatier, action of reduced nickel on halogen derivatives of fatty series in presence of hydrogen, 143
- cyclohexane and its chlorinated derivatives, 298
- direct reduction of aromatic halogen derivatives by finely-divided nickel and hydrogen, 119
- Mailard, L., constitution of the colouring-matter of indigo, 238
- Mallet, E., and P. A. Guye, atomic weights of oxygen and hydrogen, 261
- Manchot, M., formation of peroxides in the case of iron, 203
- Manganese, influences accelerating or retarding action of, considered as a metallic ferment, 12
- oxidation by means of, active influence of albumenoid matter on, 83
- titration, 83
- Manganil-manganates, alkaline earth, 155
- Manamine, 156
- Mannite and paraldehyde, combinations, 238
- Mannose, new base derived from, 156
- "Manuring of Market Garden Crops" (review), 95
- Maquenne, L., formation and saccharification of retrograde amidon, 101
- retrogradation of amidon starch, 59
- A. Fernbach, and J. Wolff, retrogradation and coagulation of amidon, 71
- and W. Goodwin, phenylurethanes of sugar, 179
- and L. Philippe, ricinine, 156
- Marc, R., decomposition of the final fractions of monazite into components, 232
- Markwald, W., contributions to our knowledge of radium, 97
- Marie, C., preparation and properties of hypophosphorous acid, 297
- and L. J. Bunel, estimation of persulphates, 113
- and R. Marquis, action of carbon dioxide on solutions of sodium nitrite, 130
- of carbonic acid on solutions of sodium nitrite, 191
- Marquis, R., and C. Marie, action of carbon dioxide on solutions of sodium nitrite, 130
- of carbonic acid on solutions of sodium nitrite, 191
- Marshall, A., vapour pressures of liquid mixtures of restricted mutual solubility, 296
- J., and J. B. Cohen, reduction of 2:6-dinitrotoluene with hydrogen sulphide, 177
- Martin, A. J., "Up to Date Tables of Imperial, Metric, Indian, and Colonial Weights and Measures" (review), 155
- G., causes of the analogy between fluorine and oxygen, 49
- connection between negative electricity and the valence of atoms, 25
- the volatility of compounds and the chemical forces at play within the molecule, 241
- Martin, G., why are some elements so much more abundant than others? 47, 118
- Maritandale, W. H., and W. W. Westcott, "Extra Pharmacopœia" (review), 309
- Maas action, lecture experiments for demonstration of, 231
- Matignon, C., action of a mixture of oxygen and hydrochloric acid on certain metals, 33
- colour reactions of vanadic acid and ethenol, 82
- and F. Bourion, preparation of anhydrous chlorides, 179
- transformation of oxides and oxygen salts into chlorides, 203
- "Matter and Energy in the Human Body, Experiments on the Metabolism of" (review), 261
- "Matter, Researches on a New Property of" (review), 155
- Matter and radio-activity, 269
- Matthey, K., constant-standard silver trial plates, 124
- Mayer, A., and V. Henri, colloidal solutions, 202
- "Medicine and Toxicology, Aide to Forensic" (review), 129
- Melior, J. W., union of hydrogen and chlorine, 152, 296
- Menthyl acetate, condensation of aldehydes with, 21
- Menthylamines, four optically isomeric, 20
- Mentrel and Gunz, M. M., action of some gases on barium ammonium, 13
- amide and nitride of barium, 34
- Mercury fulminate, estimation, 303
- vapour lamp, Bastian, 156
- Mesoxamide, oxime of, 235
- "Metabolism of Matter and Energy in the Human Body, Experiments on" (review), 261
- "Metalloids" (review), 155
- Metallurgical Analysis and Assaying" (review), 166
- "Metallurgy, Elementary Text-book of" (review), 95
- "Metals, Microscopic Analysis of" (review), 154
- Metals action of a mixture of oxygen and hydrochloric acid on, 33
- alkaline, flame spectra of, 130
- applications of theory of electrolysis to separation of from one another, 110, 118, 125
- hard and soft states in, 307
- specific heat of, 165
- Methyl acetate, saponification, 88, 103
- acetolate, 251
- alcohol, estimation in presence of ethyl alcohol, 18
- diethylacetate, preparation, 298
- tartrate, rotation value at different temperatures, 259
- Methylhydrazine, *d*-, resolution of, 19
- Methylhydrazines, *d*- and *l*-, with *d*-chlorocamphorsulphonic acid, isomeric salts of, 19
- Meunier, J., apparatus for regulating the action of vacuum pumps, 191
- history of acetals of polyatomic alcohols of sugar series, 238
- L., action of carbon dioxide on aqueous solutions of aniline in presence of nitrites, 59
- on solutions of sodium nitrite, 155
- Michaelis, A., and K. von Arend, sub-oxide of phosphorus, and solubility of red phosphorus in alcoholic aqueous potassium, 263
- Micklethwait, Frances Mary Gore, and M. O. Forster, action of nitrogen peroxide on 1-nitrocamphene, 92

- Micklethwait, Frances Mary Gore, G. T. Morgan, and H. B. Winfield, study of the substitution products of *ar*-tetrahydro-*a*-naphthylamine, 247
- Micro-manometer, 245
- Microphotography, 245
- Microscope, diffraction theory of, 215
- Microscopes, 94
- Microscopy, high power, 245
- Milk of buffalo cow, sugar in, 262
- Miller, J., and J. B. Cohen, influence of substitution in the nucleus on rate of oxidation of the side-chain, 80
- "Mineral Substances, Treatise on the Analysis of" (review), 155
- "Mineral Tables for the Determination of Minerals by their Physical Properties" (review), 190
- Minerals, action of radium rays and ultra-violet light on, 1 and substances, radio-active, 270
- radio-activity of, 133
- rare, from the Caucasus, chemical analysis of, 123
- Minet, A., electric furnace, 307
- Mingulin, J., ethylidene camphor, 168
- Mixtures, liquid, of restricted mutual solubility, vapour pressures of, 296
- viscosity of, 260
- Moissan, H., action of carbon on quicklime at temperature of fusing platinum, 119
- certain physical constants of phosphorus fluorides, 215
- "Classification des Corps Simples" (review), 225
- density of fluorine, 202
- electrolysis of calcium chloride, 297
- preparation of calcium carbide, 191
- presence of argon in the gases of the hot springs of Guadeloupe, 250
- and B. du Jassoneix, density of chlorine, 58
- and F. Siemens, action of silicon on water at a temperature of 100°, 250
- solubility of silicon in zinc and lead, 191
- Moltesster, J., and J. Ville, separation of the principles decomposing peroxide of hydrogen contained in hæmatin, 298
- Molecular weights, determining, 69
- Molecules containing the methylene group associated with negative radicals, *syntheses* effected by, 59
- Molybdates and chromates, ammoniacal double, 235
- Monazite, decomposition of final fractions of into components, 232
- Monetite, artificial production, 203
- Mono-lithium poly-meta-lanthanate, hydrated, 198
- Mononitrobenzaldehydes and hexamonic alcohols, combination, 23
- Mono-potassium poly-meta-lanthanate, hydrated, 198
- Mono-sodium poly-meta-lanthanate, hydrated, 198
- Moreau, G., thermic ionisation of saline vapours, 310
- Morgan, G. T., Frances Mary G. Micklethwait, and H. B. Winfield, study of the substitution products of *ar*-tetrahydro-*a*-naphthylamine, 247
- Morphine, reaction, 26
- Morrell, R. S., and A. E. Bellars, separation of *β*-crotonic acid from *α*-crotonic acid, 140
- and E. K. Hanson, resolution of *α,β*-dihydroxybutyric acid into its optically active constituents, 93
- Morse, F. W., report on the determination of nitrogen, 282, 291
- Moos, E. G., and C. Baskerville, contributions to the chemistry of the rare earths, 172
- Motor, petrol, projection of the indicator diagrams of, 308
- Moulin, A., colorimetric estimation of chromium, 268
- Mouneyrat, A., elimination and distribution in the organism of medicinal arsenic in form of methylarsenate of soda, 23
- influence of the chemical form under which an element is presented to the organism on the rapidity of its passage into the blood, 24
- Moureu, C., condensation of acetylenic ethers with alcohols, 107
- oxaloyl-ethylenic carbides and acids, 119
- and R. Delange, amylchloracrylic ethers, 226
- decomposition of acetylenic acids by the alkalis, 226
- general observations on the acetylenic acids, 226
- hydration of the acetylenic acids, 226
- new fatty acid,  $\gamma$ ,  $\gamma'$ ,  $\gamma''$ -trimethylbutyric acid, 226
- Mukerjee, B. M., new forms of pipettes, 161
- Murrell, W., "Aids to Forensic Medicine and Toxicology" (review), 129
- Muter, J., "Short Manual of Analytical Chemistry" (review), 47
- NAPHTHALENE**, tetrahydro-, series, 247
- Naphthylamine, *ar*-tetrahydro-*a*-, substitution products of, 247
- ar*-tetrahydro- $\beta$ -, and formaldehyde, reaction between, 247
- halogen derivatives of, 247
- a*-Naphthylmagnesium bromide, action of sulphur and selenium on, 239
- Needham, K. R., and W. H. Perkins, jun., *o*-nitrobenzoylactic acid, 70
- Neodymium, 158
- behaviour towards organic bases, 15, 27
- alum, attempt to prepare, 185
- chloride solution, saturation with hydrochloric acid, 158
- Neville, A., and R. H. Fickard, studies on optically active carbimides, 159
- Newell, L. C., "Descriptive Chemistry" (review), 257
- Nickel and hydrogen, finely divided, direct reduction of aromatic halogen derivatives of, 119
- and iron, separation, 280
- and magnesium compounds, 235
- commercial, analysis of, 45
- reduced, action on halogen derivatives of fatty series in presence of hydrogen, 143
- carbonyl, chemical reactions of, 68
- Nicardot, P., separation of chromium and vanadium, 182
- Nitrite, mercuric, and its decomposition by heat, 175
- Nitrocamphene, 1-, action of nitrogen peroxide on, 92
- Nitrogen atom, trivalent, arrangement in space of the groups combined with, 116
- atomic weight, 207
- compounds, optically active asymmetric, 69
- compressibility between one atmosphere and half an atmosphere of pressure, 86
- determination, 282, 291
- estimation, 238
- solid, densities of, 205, 217
- binoxide, action on barium-ammonium, 14
- Nitrogen peroxide, action on isonitroso-malonic ethers, 311
- on 1-nitrocamphene, 92
- sulphide, action on organic substances, 93
- Nitro-isobutylene, 23
- Nitroscamphor, 180-, 295
- Nitrostyrolene, *ar*-, reduction of, 23
- Nitroethyl chloride, action on pinene, 271
- Norton, C. L., protection of steel from corrosion, 215
- OBITUARY**, Thomas Bayley, 310
- A. W. Williamson, 257
- "Oil Fields of Russia and the Russian Petroleum Industry" (review), 120
- Oil, citronella, estimation of the adulterant in, 21, 22
- essential, of Californian laurel, constituents of, 224
- of limea, distilled, composition, 176
- Oil, thermostat for use in connection with the refractometric examination of, 80
- "Old Riddle and the Newest Answer" (review), 225
- Olein and ethers, biochemical synthesis, 130
- O'Neill, J. G., and L. M. Dennis, determination of benzene in illuminating gas, 14, 29
- "Ores, Gold and Silver, Cyaniding" (review), 249
- "Organic Substances, Optical Rotating Power of" (review), 129
- Organic bases, weak, precipitation and separation by, 43, 63, 76, 88, 103
- bodies, estimation of halogen elements in, 82
- compounds, nitrated, heat of combustion, 237
- substances, relation between the chemical composition and the density of their solutions, 117
- Organo-magnesium compounds, action on bromophenol, 262
- on phthalimide and phenylphthalimide, 244
- use in analytical operations, 139
- Osmond, F., and G. Cartaud, meteoric iron, 34
- and J. E. Stead, "Microscopic Analysis of Metals" (review), 154
- Osmosis, electric, in liquid ammonia, 59
- in methyl alcohol, 237, 286
- "Ostwald, Wilhelm" (review), 261
- Ostwald, W., elements and compounds, 220
- "Grundlinien der Anorganischen Chemie" (review), 261
- and J. H. van't Hoff, "Zeitschrift für Physikalische Chemie" (review), 167
- O'Sullivan, J., comparison of the products of the hydrolysis of potato starch with those obtained from cereal starches, 177
- Oxydases, manganous salts as in presence of a colloid, 119
- Oxidation by means of manganese, active influence of albumenoid matter on, 83
- of the side-chain, influence of substitution in the nucleus on the rate of, 80
- Oxide, bichloromethyl, symmetric, 275
- carbonic, action on barium-ammonium, 14
- compressibility between one atmosphere and half an atmosphere of pressure, 86
- decomposition in blast-furnace, 180
- Oxides, transformation into chlorides, 203
- Oxygen, action on barium-ammonium, 14
- and carbon monoxide, combining volumes of, 213
- and fluorine, causes of the analogy between, 49
- and hydrochloric acid, action of a mixture of on metals, 33
- asymmetric, 235
- atomic weight, 261
- basic properties, 295
- compressibility between one atmosphere and half an atmosphere of pressure, 86
- higher valencies of, 295
- salts, transformation into chlorides, 203
- solid, densities of, 205, 217
- Ozone, action on ethane, 273
- determining percentage in gases not dissociated by moderate heat, 201
- estimation, 243
- PCl<sub>5</sub>**, action on cyclic primary amines at boiling-point, 298
- Page, T. H., and H. E. Burgess, composition of distilled oil of limes, and a new sesquiterpene, 176
- "Paper Makers of the United Kingdom, Directory of" (review), 193
- "Paper Trade Directory" (review), 168
- Paraffins, boiling-points, 265
- Paraformaldehyde, action on sesquiterpenes, 298
- Paraldehyde and mannite, combinations, 238
- Park, J., "Cyanide Process of Gold Extraction" (review), 142
- Parsons, C. A., demonstration of the auxetophone, 245
- Patent law, Australian, 167
- "Patent Office Library, Subject-list of Works on Electricity, Magnetism, and Electro-technics in" (review), 310
- "Patents for Inventions, British, Grant and Validity of" (review), 142
- Paterson, J. W., relative effects of superphosphate and basic slag upon the feeding quality of swedes, 131
- Patterson, T. S., comparison of the rotation values of methyl-, ethyl-, and *n*-propyl-tartrates at different temperatures, 259
- influence of solvents on rotation of optically active compounds, 257
- Peachey, S. J., and W. J. Pope, preparation of tetra-alkyl derivatives of stannimethane, 20
- Pecheur, H., lead-aluminium alloys, 261
- property of tin-aluminium alloys, 286
- zinc-aluminium alloys, 274
- Pélabon, H., fusibility of mixtures of bismuth protosulphide and silver sulphide, bismuth protosulphide and antimony sulphide, 12
- mixtures of antimony triarsenide and antimony, 119
- Pencil, astigmatic, 57
- Pencils, non-homocentric, and shadows produced by them, 57, 153
- Periodides, formation in nitrobenzene solution, 273
- in organic solvents, 152
- Perkin, A. G., and A. G. Green, constitution of phenolphthalein, 141
- and F. M. Perkin, electrolytic oxidation of phenols, 92
- and E. Phipps, natural colouring matters, 18
- F. M., and A. Footenay, electrolytic oxidation of anthracene, 236

- Perkin, F. M., and W. C. Prebble, electrolytic analysis of gold, 165
- W. H., jun., experiments on the synthesis of the terpenes, 233
- ketohexahydrobenzoic acid, 141 and E. R. Needham, *o*-nitrobenzoylactic acid, 70
- and Alice Emily Smith, cis- and trans-modifications of trimethylglutamic acid, 80
- Perman, E. P., radium, 33
- Peroxide, nitric, constitution, 18
- Peroxides, formation in case of iron, 203
- Peroxyaminesulphonates, 17
- Perrin, F., and A. L. Lumière, action of chloracetamide on some aromatic amines, 208
- Persulphates, estimation, 113
- Pecci, L., organo-metallic compounds of benzoic acid, 83
- Petit, J., and A. Brochet, influence of complex ions in electrolysis by alternating currents, 143
- use of alternating currents for electrolysis, 130
- Petroleum Acids, 263
- "Pharmacy, Latin Grammar of" (review), 46
- "Pharmacy, Year-book of" (review), 21
- "Phase Rule and its Applications" (review), 201
- Phase rule, application to precipitation of colloids, 202
- Phenol and diazobenzene, limit of union, 311
- tri-iodine, 34
- Phenolic compounds, bromination, 272
- Phenolphthalein, behaviour in presence of bicarbonates and of alkaline carbonates, 27
- constitution, 141
- Phenols, dinaphthopyranic, 119
- electrolytic oxidation, 92
- Phenyl carbonate, chloruration in presence of antimony chloride, 251
- in presence of iodine, 238
- Phenyldimethylallylammonium compounds, 177
- Phenylhydrazine, action on oxidised compounds of selenium and tellurium, 131
- and cerium, 64
- and ferric salts, 65
- and thorium, 64
- hydrochloride, hydrolysis, 77
- Phenyl migration, 22
- Phenylmagnesium bromide, action on anthraquinone, 130
- of sulphur and selenium on, 239
- Phenylphthalimide, action of organo-magnesium compounds on, 244
- Philippe, L., and L. Maquenne, ricinine, 156
- Phillips, C. E. S., automatic gas pump, 213
- vacuum pump, 308
- Phipps, E., and A. G. Perkin, natural colouring matters, 16
- Photographic transparencies, three-colour, illumination by spectrum colours, 212
- Phosphate, dicalcic, 203
- methyl-amino-magnesian, 34
- trimethylamino-magnesian, 34
- Phosphates, amino-magnesian, 34
- Phospho-nitrated bases, 215
- Phosphorescent materials, 245
- Phosphorus and sulphur, 54
- compounds, organic, 116
- red, solubility in alcoholic aqueous potash, 263
- fluorides, physical constants of, 215
- sesquisulphide and sulphur, action of heat and light on mixtures of in carbon disulphide solution, 130
- sub-oxide, 205
- sulphides, formation in the cold, 130
- Phthalimide, action of organo-magnesium compounds on, 244
- Physical Society, 57, 93, 127, 133, 177, 212, 248, 264, 306
- Address to, 93
- Physotype, 308
- Pickard, R. H., and A. Neville, optically active carbimides, 259
- Pilling, normal, as connected with Osborne Reynolds's theory of the universe, 212
- Pinacolin and pinacone, reactions of, 35
- Pineane, action of nitrosyl chloride on, 271
- Piperidine urethanes, phenolic, 238
- Pipettes, 161
- Planes, F., colorimetric estimation of bismuth, 10
- Plant, indigo, fermentation, 296
- Plants, chlorine in, 33
- Platinocyanides, preparing, 274
- Platinum solution, electrolytic, 274
- Plimmer, R. H. A., separation and estimation of silver cyanide and silver chloride, 19
- Polarisation, cathodic, 239
- Pollard, C. E., colouring varnish, 264
- Pollak, J. H., heat of formation of glucinum chloride, 176
- Pope, W. J., and S. J. Peachey, preparation of tetra-alkyl derivatives of stannimethane, 20
- Porcher, C., sugar in milk of buffalo cow, 262
- and M. Brissac, attempts to make aniline and urea take the form of amino-magnesian phosphate, 35
- some amino-magnesian phosphates, 34
- Potash, alcoholic aqueous, solubility of red phosphorus in, 263
- caustic, fusion of isopilocarpine with, 81
- determination, 34
- Potassium ferrocyanide and potassium ferricyanide, chemical equilibrium between in presence of alkalis, 254
- iodide, aqueous, nature of a solution of iodine in, 176
- permanganate and oxalic acid, speed of reaction between, 96
- Potter, H., biochemical synthesis of olein and certain ethers, 130
- Power, F. B., and F. H. Gornall, constituents of chaunmoogra seeds, 294
- constitution of chaunmoogric acid, 295
- gynocardin, 295
- and F. H. Lees, constituents of essential oil of Californian laurel, 224
- and F. Tutin, levorotatory modification of quercitol, 223
- Pozzi-Escot, E., colour reactions of molybdic acid, 106
- Praseodymium, 147
- behaviour toward organic bases, 15, 27
- alum, attempt to prepare, 184
- citrate, 148
- Prebble, W. C., and F. M. Perkin, electrolytic analysis of gold, 165
- Precipitation and separation by weak organic bases, 43, 63, 76, 88, 103
- Printing, application of thin-film electrolysis to, 165
- Procter, H. K., "Principles of Leather Manufacture" (review), 166
- n*-Propyl, ethyl, and methyl tartrates, rotation values at different temperatures, 239
- Propylpyrocatechin carbonate, 143
- Prud'homme, M., chemical equilibrium between ferro- and ferricyanide of potassium in presence of alkalis, 254
- between ferro- and ferri-hydrocyanic acids, 299
- Pump, gas, automatic, 213
- vacuum, automatic, 308
- Pumps, vacuum, apparatus for regulating action of, 191
- Pyridic bases, action of bromosuccinic and dibromosuccinic acid on, 34
- QUERCITOL**, levorotatory modification of, 223
- Quicklime, action of carbon on at the temperature of fusing platinum, 119
- Quinolic bases, action of bromosuccinic and dibromosuccinic acid on, 34
- RADIATION**, Röntgen, energy of secondary, 178
- Radicles, hydroxyl, estimation, 19
- Radio-active minerals and substances, 270
- "Radio-activity" (review), 309
- Radio-activity and matter, 289
- induced, 97
- of bacteria, 245
- of minerals and mineral waters, 133
- "Radium and All About It" (review), 250
- Radium, 35, 97, 174
- and barium, separation, 97
- emanation, spectrum of, 301
- production of helium from, 255, 269
- rays, action on halides of alkali metals, 246
- on minerals, 1
- photographic action of, 58
- what may result from the study of, 47
- barium chloride, anhydrous, phosphorescence of, 97
- bromide, examination of sample of gas occluded in, 85
- Ramage, H., boiling-points of homologous compounds, 265
- distribution and spectra of metallic vapours in electric sparks, 265
- Ramsey, Sir W., "Introduction to the Study of Physical Chemistry" (review), 134
- and J. N. Collie, spectrum of radium emanation, 301
- and F. Soddy, further experiments on production of helium from radium, 255, 266
- Rape, green, distribution of sulphur in, 35
- Raper, H. S., J. T. Thompson, and J. B. Cohen, action of sodium hypochlorite on aromatic sulphonamides, 153
- Ray, P. C., mercuric nitrite and its decomposition by heat, 175
- Rayleigh, Lord, compressibilities of oxygen, hydrogen, nitrogen, and carbonic oxide between one atmosphere and half an atmosphere of pressure, 86
- Red, Venetian, preparing, 197
- "Refuse disposal and Power Production" (review), 285
- Rengade, E., action of carbonic anhydride on ammonium metals, 179
- "Report on Agricultural Work in Botanic Gardens and Government Laboratory at British Guiana" (review), 274
- Report of International Committee on atomic weights, 68, 164, 173
- on ash, 41, 54
- on determination of nitrogen, 282, 291
- Reverdin, F., and P. Crepieux, nitration of acetylgascol, 287
- Reynolds, O., normal piling as connected with theory of the universe of, 212
- Richards, J. W., thermo-chemistry of theory of electrolytic dissociation, 31, 37
- and V. Englehardt, "Electrolysis of Water" (review), 214
- Ricinine, 156
- Rimini, E., new reaction for aldehydes, 131
- Ringer, W. E., mixed crystals of sulphur and selenium, 131
- Ripple marks, origin and growth, 308
- Rivals, P., and H. Saubigny, separation of iodine from alkaline halogen salts of iodine, chlorine, and bromine, 12
- Rivot's method of estimation of iron in presence of zirconium, 311
- Roberts, J., "Grant and Validity of British Patents for Inventions" (review), 142
- W., and J. J. Sudborough, di-ortho-*o*-substituted benzoic acids, 19
- Robin, L., new indicator, 262
- Roe, S., camphor monopoly in Japan, 250
- Rohn, E., and A. Gutbier, estimation of selenium, 311
- Romyn, G., and J. A. Voorthuis, estimation of formic aldehyde in air, 23
- Röntgen radiation, energy of secondary, 178
- Roseco, Sir H. E., Address to, 222
- Rose, T. K., properties of silver-cadmium series of alloys, 86
- Roux, E., mannamine, 156
- Royal Institution, 24, 58, 71, 95, 130, 131, 143, 179, 192, 209, 220, 239, 251, 266
- Society conversazione, 245, 308
- Rubidium, atomic weight, 223
- Ruff, O., and G. Winterfeld, bromides of sulphur, 287
- Rubemann, S., and E. K. Watson, contributions to knowledge of  $\beta$ -diketones, 140
- Rupp, E., iodometric estimation of thallium, 24
- iodometry of the peroxides of calcium, strontium, barium, magnesium, and sodium, 96
- and L. Kraus, iodometric estimation of copper by means of xanthate of potassium, 288
- and O. Schaumann, iodometric estimation of bismuth in the form of chromate, 143
- "Russian Oil Fields, and the Russian Petroleum Industry" (review), 120
- Rutherford, E., "Radio-activity" (review), 309
- SABATIER**, P., and A. Mailhe, action of reduced nickel on alkyl halogen derivatives of fatty series, 143
- cyclohexane and its chlorinated derivatives, 298
- direct reduction of aromatic halogen derivatives of finely-divided nickel and hydrogen, 119
- and J. B. Senderens, direct hydrogenation of aniline, 155
- of homologues of aniline, 310
- preparation of cyclohexanol and cyclohexanone from phenol, 33
- Saccharine, detection by new reactions, 132
- Saccharose and metallic salts, compounds, 59, 179
- Sack, M., formation of alloys of sodium and their importance in phenomena of cathodic polarisation, 339
- St. Bogdan, M., use of binocide of lead in analysis, 26
- Sakurai, J., and K. Ikeda, international atomic weights, 305
- Salt of manganous and tungstic acids, complex double, 288
- Salter, C. T. C., and Dr. von Schwartz, "Fire and Explosion Risks" (review), 286
- Salts, carbonatopentamine cobaltic, 275
- dinaphthopyryl, union with dialcoholic aromatic amines, 168

- Salts, ferric, and phenylhydrazine, 65  
 formation from diortho-substituted benzoic acids and organic bases, 19  
 isomeric, of *d*- and *l*-methylhydrazines with *d*-chloroamphorsulphonic acid, 19  
 manganous, as oxydases in presence of a colloid, 119  
 metallic, and saccharose, compounds, 59, 179  
 of lower fatty acids, solubility, 193  
 oxygen, transformation into chlorides, 203  
*d*- and *l*-phenylbenzylmethyl-ethylammonium, 69  
 silver and lead, of monoacylphosphoric acids, 203  
 Salway, A. H., and F. S. Kipping, arrangement in space of groups combined with trivalent nitrogen atom, 116  
 Samaria, preparation, 277  
 Samarium, 234  
 atomic weight, 277  
 Sand, H. J. S., and J. E. Hackford, electrolytic estimation of minute quantities of arsenic, 272  
 Sazerc, R., oxidising bacterium, 287  
 Schenk, R., and P. Zimmermann, decomposition of carbonic oxide in blast-furnace, 180  
 Scott, A., combining volumes of carbon monoxide and oxygen, 223  
 vapour density of hydrazine hydrate, 223  
 Sea-salt, solubility of hydrated sulphate of lime in solutions of, 9  
 Seeds, chaumogra, constituents, 294  
 Selenium, action on bromides of phenyl-magnesium and  $\alpha$ -naphthyl-magnesium, 239  
 on organo-magnesium compounds of mono- and di-halogen aromatic hydrocarbons, 231  
 and sulphur crystals, mixed, 131  
 and tellurium, oxidised compounds of, action of phenylhydrazine on, 131  
 colloidal, aqueous solutions of, 143  
 estimation, 311  
 Senderens, J. B., and P. Sabatier, direct hydrogenation of aniline, 155  
 of homologues of aniline, 310  
 preparation of cyclohexanol and cyclohexanone from phenol, 53  
 Sensitometers, colour, calculation of colours for, 212  
 Sesquiterpene, composition of a new, 176  
 Sesquiterpene, action of para-formaldehyde on, 298  
 Sexton, A. H., "Elementary Text-book of Metallurgy" (review), 95  
 Seyewitz, A., and M. Gibello, new polymers of formaldehyde, 298  
 synthesis of sugars from trioxymethylene and sodium sulphite, 86  
 Shadows produced by axially symmetrical pencils possessing spherical aberration, 153  
 Shafts, whirling and transverse vibrations of, 153  
 Sherrill, P. E., and C. A. B. Garrett, coherence and recombination, 285  
 Sheridan, F., physiology, 308  
 Sherman, H. C., sulphur and phosphorus, 54  
 Shrivell, F. W. E., and B. Dyer, "Manuring of Market Garden Crops" (review), 95  
 Side-chain, influence of substitution in nucleus on the rate of oxidation of, 80  
 Siemens, F., and H. Moissan, action of silicon on water at a temperature of 100°, 250  
 solubility of silicon in zinc and lead, 191  
 Silberrad, O., action of ethyl  $\beta$ -iodo-propionate on ethyl disodioethane-tetracarboxylate, 176  
 and T. H. Easterfield, ethyl carboxylglutarate, 259, 296  
 Silica, colorimetric, determination of small quantities, 73, 89, 101  
 Silicates, alkaline and alkaline-earthly, and silicic acid, 23  
 reduced, 235  
 Silicon, action on water at a temperature of 100°, 250  
 derivatives, organic, 81  
 reduction by hydrogen, 274  
 solubility in zinc and lead, 191  
 volatilisation in hydrogen, 286  
 Silk, H., J. T. Hewitt, and J. Kenner, bromination of phenolic compounds, 272  
 "Silver and Gold Ores, Cyaniding" (review), 249  
 Silver, colloidal, preparation and properties of pure, 218  
 trial-plates, constant standard, 124  
 cadmium series of alloys, properties of, 86  
 chloride and silver cyanide, separation and estimation, 19  
 nitrate and ammonia, compounds, 275  
 salts of monoacylphosphoric acids, 203  
 sulphide and bismuth protosulphide, fusibility of mixtures, 12  
 Simmonds, C., reduced silicates, 235  
 Simon, L. J., diurides, 130  
 glyoxylic ureides, 143  
 homoallantoic ether, 130  
 new action of hydroxylamine, 22  
 and A. Conduche, new reaction of aldehydes, 251  
 Simonet, A., combination of hexatomic alcohols with mononitrobenzaldehydes, 23  
 and L. Vignon, action of diisobenzene chloride on diphenylamine, 275  
 Skinner, S., photographic action of radium rays, 58  
 Slag, basic, and superphosphate, relative effects upon the feeding quality of swedes, 131  
 Smart, H., and H. F. Julian, "Cyaniding Gold and Silver Ores" (review), 249  
 Smith, A., "Laboratory Outline of General Chemistry" (review), 154  
 A., and R. Kerr, microphotography, 245  
 Alice Emily, and W. H. Perkin, jun., cis- and trans-modifications of trimethylglutaconic acid, 80  
 C., tetrahydronaphthalene series, 247  
 N., gaseous carbon monosulphide described by Thomsen, 25  
 Soda carbonate to precipitate lime and magnesia for chemical purification of water, 254  
 nitroprussiate, toxicity of, 226  
 Soddy, F., and Sir W. Ramsay, further experiments on production of helium from radium, 255, 266  
 Sodeau, W. H., estimation of unburnt products in chimney gases, 61  
 Sodium alloys, formation, 239  
 acetylacetone, action of epichlorhydrine on, 59  
 carbonate, ignition with, 56  
 hydroxide, fusion with, 56  
 hypochlorite, action on aromatic sulphonamides, 153  
 methoxide, action on benzophenone chloride and benzylidene chloride, 272  
 Sodium monosulphide as an indicator in estimation of glucose by Fehling solution, 71  
 nitrite solutions, action of carbon dioxide on, 130, 155  
 of carbonic acid on, 191  
 percarbonate, 168  
 peroxide, iodometry of, 96  
 succinate, condensation of furfuraldehyde with, 80  
 sulphite and trioxymethylene, synthesis of sugars from, 86  
 Soil analysis, rapid, 183  
 Soils, scale for chemical valuation, 197  
 Solids, formation at low temperatures, 145  
 "Solution, Treatise on the Theory of" (review), 191  
 Solutions, colloidal, 202  
 in antimony sulphide, cryoscopic examination of, 311  
 Solvents, influence on rotation of optically active compounds, 297  
 Sommelet and Behal, MM., method of synthesis of aldehydes, 83  
 Sorel, E., "La Grande Industrie Chimique Minérale" (review), 46  
 Southerton, F., conversion of isopropyl alcohol into isopropyl ether by sulphuric acid, 261  
 separation of iron and chromium, 183  
 Sowter, R. J., portable electroscope, 128  
 Sparks, electric, distribution and spectra of metallic vapours in, 253  
 Specific heat of metals, 165  
 relation to atomic weight, 165  
 Spectra, absorption, of solutions of salts of didymium containing phosphoric acid, 299  
 flame, of the alkaline metals, 130  
 of metallic vapours in electric sparks, 253  
 Spectrum of the radium emanation, 301  
 of zinc, 251  
 spark and arc, of zinc, invariability of the wave lengths in, 98  
 Spencer, A., and J. W. Walker, compounds of aluminium chloride with organic substances containing oxygen, 294  
 J. F., and A. W. Titherley, condensation of furaldehyde with sodium succinate, 80  
 Spice, M., detection of saccharine by new reactions, 132  
 Sprankling, C. H. G., W. A. Bone, and J. J. Sudborough, acid esters of methylsuccinic acid, 177  
 Stachyose, 287  
 Staehelin, R., part played by benzene in poisoning by coal-gas, 74  
 Stanley, H., solubility of some salts of lower fatty acids, 193  
 Stannimethane, preparation of tetra-alkyl derivatives of, 20  
 Starch, amidon, retrogradation of, 59  
 hydrolysis by diastase, 247  
 potato, comparison of products of hydrolysis of with those obtained from cereal starches, 177  
 composition, 132  
 Stead, J. E., and F. Osmond, "Microscopic Analysis of Metals" (review), 154  
 Steel, protection from corrosion, 215  
 Steels, determining the critical points of, 34  
 nickel, allotropic transformations of, 130  
 silicon, constitution and properties, 34  
 vanadium, constitution and properties, 130  
 Stern, A. L., so-called hypercellulose, 117  
 Stevenson, R., and C. Baskerville, contributions to the chemistry of the rare earths, 158  
 Stockings, W. E., and W. A. Bone, slow combustion of ethane, 246  
 and J. Drugman, action of hydrogen sulphide on formaldehyde and acetaldehyde solutions, 260  
 Stoeker, M., and F. Haber, "Praktische Übungen zur Einführung in die Chemie" (review), 154  
 Stoebling and Guyot, MM., certain derivatives of tetramethyldiaminophenylloxanthranol, 107  
 Stolzenberg system of keeping notes, 269  
 Storbeck, O., and G. Bodlander, study of cuprous compounds, 59  
 Stoves, germination, application of acetylene gas to heating of, 215  
 Stresses in a magnetostatic field, 128  
 Strontium peroxides, iodometry of, 96  
 Strutt, R. J., radio-activity of minerals and mineral waters, 133  
 Sudborough, J. J., and H. Hibbert, estimation of hydroxyl radicals, 19  
 and W. Roberts, diortho-substituted benzoic acids, 19  
 W. A. Bone, and C. H. G. Sprankling, acid esters of methylsuccinic acid, 177  
 Sugar in milk of buffalo cow, 262  
 inversion, 156  
 phenylurethanes, 179  
 Sugars from trioxymethylene and sodium sulphite, synthesis, 86  
 Sulphate, cuproscopic formation and solubility, 224  
 Sulphates, determination, 54  
 double, 172, 184  
 Sulphides, colloidal, examination of, 59  
 Sulphonamides, aromatic, action of sodium hypochlorite on, 153  
 Sulphur, action on bromides of phenylmagnesium and  $\alpha$ -naphthylmagnesium, 239  
 on organo-magnesium compounds, 227  
 of mono- and di-halogen aromatic hydrocarbons, 231  
 and phosphorus, 54  
 sesquisulphide, action of heat and light on mixtures of in carbon disulphide solution, 130  
 and selenium crystals, mixed, 131  
 and tellurium compounds, 131  
 determination, 41, 54  
 in green rape, distribution, 55  
 bromides, 287  
 Superphosphate and basic slag, effects upon feeding quality of swedes, 131  
 Sutherland, W. G., "Dispensing made Easy" (review), 142  
 Sweden, baumanites from, 211  
 Swedes, effects of superphosphate and basic slag upon the feeding quality of, 131  
 Swinburne, J., "Entropy" (review), 225  
 Syntheses by means of molecules containing methylene group associated with negative radicals, 59  
 "TABLES, Calculating" (review), 214  
 "Tables of Imperial, Metric, Indian, and Colonial Weights and Measures, Up to Date" (review), 155

- Taboury, F., action of sulphur and selenium on bromide of phenylmagnesium and bromide of  $\alpha$ -naphthylmagnesium, 238  
on organo-magnesium compounds of mono- and di-halogen aromatic hydrocarbons, 231
- Tanatar, S., percarbonate of sodium, 168
- Tannin and bismuth compounds, 238
- Tanret, C., stachyose, 287
- Tar distilleries, accidents in, 35
- Tartrate, crystalline chromous, 297
- Tartrates, optical activity in aqueous solution, 287
- Tattersall, G., isomeric salts of  $d$ - and  $l$ -methylhydramines with  $d$ -chlorocamphorsulphonic acid, 19  
resolution of  $dl$ -methylhydramine, 19
- Taylor, Millicent, and F. E. Francis, additive products of benzylideneaniline with ethyl acetate and ethyl methylacetate, 259
- Tchernik, G. P., chemical analysis of two rare minerals from the Caucasus, 123
- Tchougacoff, L. A., use of organo-magnesian compounds in analytical operations, 139
- Technical research and the college system, 230
- "Technica" (review), 240
- Telephone cable, propagation of alternating current along, 306
- Tellurium, 174  
and antimony, separation, 168  
and selenium, oxidised compounds of, action of phenylhydrazine on, 131  
and sulphur compounds, 131  
estimation, 143
- Temperature, action on period of induction, 152
- Terpene, synthesis of, 223
- Terpin hydrate, synthesis of, 223
- Terpineol, inactive, synthesis of, 223
- Tetrahydronaphthalene series, 247  
or Tetrahydro- $\alpha$ -naphthylamine, substitution products of, 247
- Tetramethyldiaminophenylloxanthranol derivatives, 107
- Tetramethylene series, synthesis in, 251
- Thallium estimation, 24, 311
- Thermostat for use in connection with refractometric examination of oils and fats, 80
- Thibault, P., action of hydrated oxide of bismuth on acid isomers of gallic acid, 227  
compounds of bismuth and tannin, 238  
some compounds by means of which the constitution of bismuthogallic acid may be determined, 23
- Thiel, A., simplification of the method for estimation of zinc in the form of sulphide, 16
- Thiobenzoylacetone, 23
- Thiocarbamide, 141
- Thiourethane, picryl derivatives, 235
- Thompson, A. B., "Oil Fields of Russia and the Russian Petroleum Industry" (review), 120
- Thompson, J. T., H. S. Raper, and J. B. Cohen, action of sodium hypochlorite on the aromatic sulphonamides, 153
- W. P. and Co., Australian Patent law, 167
- Thomsen, M., gaseous carbon monosulphide described by, 25
- Thorium and beryllium, separation, 77  
and phenylhydrazine, 64  
behaviour toward organic bases, 15, 27
- Thorpe, T. E., interdependence of the physical and chemical criteria in analysis of butter fat, 80  
thermostat for use in connection with refractometric examination of oils and fats, 80  
and J. Holmes, estimation of methyl alcohol in presence of ethyl alcohol, 18
- Threlfall, R., micro-manometer, 245
- Tiffeneau, M., phenylic migration, 22
- transformation of primary  $\alpha$ -glycols into the corresponding aldehydes, 59
- two isomeric  $\beta$ -methylcinnamic acids, 251
- Tilden, W. A., action of nitrosyl chloride on pinene, 271  
Address to Chemical Society, 190  
specific heats of metals, and relation of specific heat to atomic weight, 165  
"Times Engineering Supplement" (review), 251
- Tin-aluminium alloys, property of, 286
- Tinkler, C. K., J. J. Dobbie, and A. Lauder, relative strengths of the alkaline hydroxides and of ammonia as measured by their action on cotaniline, 17
- Titanium and beryllium, separation, 76  
and iron, separation, 63
- Titherley, A. W., and J. F. Spencer, condensation of furfuraldehyde with sodium succinate, 80
- "Tokyo Imperial University of Calendar" (review), 249
- Toluenes, mono- and dichloro-, oxidation, 80
- Tommassi, D., law of thermic constants, and heat of formation of compounds of barium, 123  
new electric accumulator, 287
- "Toxicology and Medicine, Aids to Forensic" (review), 129
- Travers, M. W., formation of solids at low temperatures, 145
- Trials-plates, constant-standard silver, 124
- Trillat, A., active influence of albumenoid matter on oxidation by means of manganese, 83  
influences accelerating or retarding action of manganese considered as a metallic ferment, 12  
manganous salts as oxydases in presence of a colloid, 119
- Trioxymethylene and sodium sulphite, synthesis of sugars from, 86
- Triphenylmethane colouring matters, 297
- Truffa t, G., and A. Hébert, chemical study of the culture of chrysanthemums, 60
- Tube, safety, new, 262
- Turpentine essence from Landes, estimation, 257
- Turrentine, J. W., and C. Baskerville, contributions to chemistry of rare earths, 147
- Tutin, F., and F. B. Power, levorotatory modification of quercitol, 223  
and F. S. Kipping, four optically isomeric  $l$ -menthylamines and their salts, 20
- ULKE, T., and V. Engelhardt, "Die Elektrolytische Raffination des Kupfers" (review), 225
- Umbellulone derivatives, 224
- Universes, theory of, normal piling as connected with, 212
- Unruh, V., constants of sulphide of carbon, 83  
and M. Erdmann, yellow arsenic, 71
- Uranium nitrate, precipitation of alkaloïds by, 26
- Urban, G., and H. Lacombe, europium, 179  
preparation of samaria and atomic weight of samarium, 277  
use of bismuth as a means of separation in the rare earth series, 52
- Urea, attempt to make take the form of amino-magnesian phosphate, 35
- Ureides, glyoxylic, 143
- Urethane, picryl derivatives, 235
- Urethanes, phenolic, of piperidine, 238
- VACUUM drying machine, 312
- machinery, 288
- Vaillant, V., thiobenzoylacetone, 23
- Valenta, E., and J. M. Eder, invariability of the wave lengths in the spark and arc spectrum of zinc, 98
- Van't Hoff, J. H., and W. Ostwald, "Zeitschrift für Physikalische Chemie" (review), 167
- Vanadium and chromium, separation, 182
- Vapour pressures of liquid mixtures of restricted mutual solubility, 206
- Vapours, metallic, in electric sparks, distribution and spectra, 253
- saline, thermic ionisation, 310
- Varenne, E., and L. Godefroy, ethylic alcohol hydrates, 22  
hydrates of methylic alcohol and acetone, 244
- Varnish, colouring, 264
- Vegetable acidity, influence of its surroundings on, 227
- Veitch, F. P., colorimetric determination of small quantities of phosphoric acid and of silica, 73, 89, 101
- Venetian red, preparing, 197
- Ventilation of the House of Commons, 168
- Vezeas, M., estimation of essence of turpentine from Landes, 287
- Viard, M., and A. Kling, differentiation of primary, secondary, and tertiary alcohols of fatty series, 287
- Vibrograph, 245
- Vignon, L., determination of carbonate of sodium necessary to precipitate lime and magnesia for chemical purification of water, 254  
influence of copper on silvicing of glass, 23  
limit of the union of diazobenzene and phenol, 311  
and A. Simonet, action of diazobenzene chloride on diphenylamine, 275
- Vigouroux, E., formation of hydrogen allicide, 286
- Vigreux, H., washer and safety tube, 262
- Ville, J., and J. Molesier, separation of principles decomposing peroxide of hydrogen contained in hæmatis, 298
- Viscosity, 236  
of liquid mixtures, 260  
Volatility of compounds and chemical forces at play within the molecule, connection between, 241
- Von Aренд, K., and A. Michaelis, suboxide of phosphorus and solubility of red phosphorus in alcoholic aqueous potash, 263
- Von Dielerich, A., and L. Wohler, lecture experiments for the demonstration of mass action, 231
- Von Schwarz, Dr., and C. T. C. Salter, "Fire and Explosion Risks" (review), 286
- Voorthuis, J. A., and G. Romyn, estimation of formic aldehyde in the air, 23
- WADMORE, J. M., and F. D. Chattaway, derivatives of highly-substituted anilines, 81
- Wagner, A., absorption spectra of solutions of salts of didymium containing phosphoric acid, 299
- Wahl, A., and L. Bouveault, action of peroxide of nitrogen on isonitrosomalonic ethers, 311  
gradual synthesis of the fatty aldehydes, 226  
nitro-isobutylene, 23  
preparation of diketonic ethers, 268  
reduction of  $\alpha$ -nitrostyrolene, 23
- Walcott, C. D., radio-active minerals and substances, 270
- Walden, P., "Wilhelm Ostwald" (review), 261
- Walker, C., and W. A. Macleod, "Metallurgical Analysis and Assaying" (review), 166
- G. W., stresses in a magnetostatic field, 128
- J., "Introduction to Physical Chemistry" (review), 214
- J. W., ionisation and chemical combination, 294  
and A. Spencer, compounds of aluminium chloride with organic substances containing oxygen, 294
- D. McIntosh, and E. H. Archibald, ionisation and chemical combination in the liquefied halogen hydrides and hydrogen sulphide, 294
- Washer, 262
- Wassermann, A., and C. Bolduan, "Immune Sera" (review), 310
- "Water, Electrolysis of" (review), 214
- Water at a temperature of 100°, action of silicon on, 250
- Boottle well, composition, 9  
estimation of organic matter in, 219, 229  
purification by continuous fractional distillation, 141  
determination of carbonate of sodium necessary to precipitate the lime and magnesia for, 254  
supply, London, 32, 88, 146, 211, 242, 304
- Waters containing chlorides and bromides, estimation of organic matters in, 219  
mineral, radio-activity of, 133  
potable, in South-West Lancashire, 6
- Watson, E. R., and S. Ruhemann, contributions to knowledge of diketones, 140
- W., hints on the preparation of diagrams, 128  
quartz-thread, vertical force magnetograph, 127
- "Weights and Measures, Up to Date Table of Imperial, Metric, Indian, and Colonial" (review), 115
- Weights, atomic, report of International Committee, 68  
molecular, determining, 69
- Werner, A., and N. Goaling, carbonatopentamine cobaltic salts, 275
- "Werner's, Alfred, Theory of Carbon Atom, and Stereochemistry of Carbocyclic Compounds" (review), 46
- Westcott, W. W., and W. H. Martindale, "Extra Pharmacopœia" (review), 399
- Whetham, W. C. D., "Treatise on the Theory of Solution" (review), 191
- Whiteley, Martha Annie, oxime of mesoxamide, and allied compounds, 235



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|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <p>Williamson, A. W. (obituary), 237<br/>         Winfield, H. B., G. T. Morgan, and Frances Mary Gore Michaelthwait, substitution products of <i>α</i>-tetrahydro-<i>β</i>-naphthylamine, 247<br/>         Winkler, C., radio-activity and matter, 289<br/>         Winterfield, G., and O. Ruff, bromides of sulphur, 287<br/>         Wöhler, L., and A. von Dieterich, lecture experiments for demonstration of mass action, 231</p> | <p>Wolff, J., L. Maquenne, and A. Fernbach, retrogradation and coagulation of amidon, 71<br/>         Wood, R. W., cases of interference and diffraction, 57<br/>         Wood treated to resist fire, testing, 32, 40<br/>         Wright, A. E., apparatus for measuring contents of human blood, 245<br/>         Wuyts, H., and G. Coeyns, action of sulphur on organo-magnesian compounds, 227</p> | <p>"X-RAYS Simply Explained" (review), 190<br/>         YOUNG, S., "Fractional Distillation" (review), 129<br/>         Youts, L. A., methods for estimation of antimony, 299<br/>         ZINC in form of sulphide, estimation, 16<br/>           spectrum, 251<br/>           spark and arc, invariability of wave-lengths in, 98</p> | <p>Zinc aluminium alloys, 274<br/>         peroxides, 95<br/>         Zimmermann, H., "Calculating Tables" (review), 214<br/>         P., and R. Schenk, decomposition of carbonic oxide in the blast-furnace, 180<br/>         Zirconium and beryllium, separation, 77<br/>         and iron, separation, 63<br/>         behaviour toward organic bases, 15, 27</p> |
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END OF VOLUME LXXXIX.











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